RIA Report 52-2732

THE DEPARTMENT OF DEFENSE
MILITARY RUBBER LABORATORIES

LOW TEMPERATURE TESTING OF ELASTOMERS CONFERENCE

SPONSORED BY THE
ORDNANCE CORPS - DEPT. OF THE ARMY

4 and 5 March 1952
The Pentagon, Washington, D. C.

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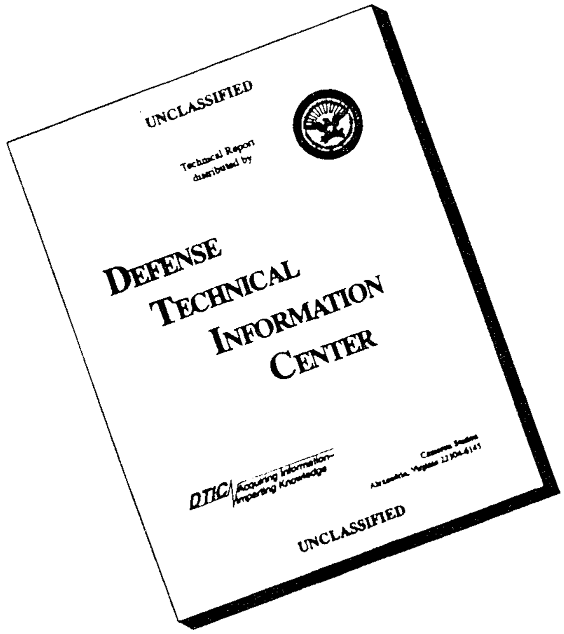

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PREFACE

The determination of the physical characteristics of elastomers at low temperatures has not been standardized within the rubber industry because the many and varied uses of rubber preclude the use of one specific test method.

The standardization of test equipment and procedures for specification testing, and Research and Development Evaluation, has not progressed sufficiently by the rubber industry or standardizing agencies such as the Society of Automotive Engineers or the American Society for Testing Materials, to insure adequate low temperature performance.

Since a large proportion of rubber goods is currently being manufactured for the military services, and since low temperature performance is mandatory, it becomes apparent that the military laboratories should take the lead in establishing adequate procurement specifications.

It was for this purpose, that the Conference on low temperature testing of elastomers was held.

AGENDA OF
LOW TEMPERATURE RUBBER CONFERENCE
4 and 5 MARCH 1952
Room 5C636, The Pentagon
Washington 25, D. C.

TUESDAY 4 MARCH 1952

Irving Kahn, Presiding Officer, Army Ordnance

9:00 AM	Introductory Remarks	- Col. A. R. Del Campo (Chief, Res & Mat'ls Br, OCO)
9:10 AM	Status of Low Temperature Test Methods in Proposed Revision of Federal Specification ZZ-R-601	- R. F. Tener (National Bureau of Standards)
9:20 AM	Broadscale Objectives of the low temperature test methods work of Bureau of Ships	- T. A. Werkenthin (Bureau of Ships)
9:40 AM	Review of the "High Lights" of Low Temperature Test Work of Bureau of Ships	- J. Lichtman or C. Chatten (New York Naval Shipyard)
10:30 AM	General Review of low temperature Test work at Government laboratories, Akron, Ohio	- B. Labbe and Dr. Helin (Government Laboratories)
11:30 AM - 12:30 PM	LUNCH, Pentagon Cafeteria	
12:30 PM	Review of the status of SAE-ASTM subsection IV-L findings on low temperature tests	- Dr. Hanson (Rock Island Arsenal)
1:00 PM	Review of ASTM Task Group B of E-1 work concerning flexibility tests	- M. Boor (Quartermaster Corps)
1:30 PM	Review of low temperature test work of the Corps of Engineers with emphasis on tests in the proposed uncoordinated Military specification for Cable, Power, Electric	- P. Mitton or Dr. French Corps of Engineers.
2:30 PM	General discussion and review of low temperature test work of Bureau of Aeronautics	- R. Harper (Naval Air Experimental Station)

AGENDA OF LOW TEMPERATURE RUBBER CONFERENCE 4 March 1952

3:00 PM	General discussion of low temperature test work at Wright Air Development Center	- Lt. Bernstein (WADC)
3:30 PM	General review of low temperature test work of Army Ordnance	- R. Shaw (Rock Island Arsenal)
4:00 PM	General review of low temperature test work of Quartermaster Corps	- C. Griffis (Quartermaster Corps)

WEDNESDAY 5 MARCH 1952

9:00 AM	General review of the low temperature work of other agencies.
10:00 AM	Summary of the needs of each represented activity with suggestions from others in attendance.
12:00 to 1:00	LUNCH, Pentagon Cafeteria
1:00 PM	Selection and choosing of standard low temperature test methods and apparatus.
3:00 PM	Plans for future cooperation and action.

ATTENDANCE LIST FOR
LOW TEMPERATURE RUBBER CONFERENCE

Tuesday 4 March 1952

Lt. Cmdr. W.B. Heidt, Jr.	BuShips, Navy, Washington 25, D. C.
J. E. Gaughan	Detroit Arsenal, Center Line, Michigan
C. R. Strong	Detroit Arsenal, Center, Line Michigan
F. E. Rupert	ERDL, Fort Belvoir, Va.
C. B. Griffis	OQMG, Phila, Pa.
Sgt. R. H. Brown	Chemical Corps, Maryland
Lt. Jos. H. Bernstein	WADC, Wright-Patterson AF Base, Dayton, Ohio
J. Lifland	Signal Corps, Engrg Labs, Ft Monmouth, NJ
P. Lichtenstein	Signal Corps, Engrg Labs, Ft Monmouth, NJ
E. F. Greenleaf	BuShips, Navy, Washington 25, D. C.
J. Z. Lichtman	Materials Lab, NY Naval Shipyard, Brooklyn
T. A. Werkenthin	BuShips, Navy, Washington 25, D. C.
B. G. Labbe	Univ. of Akron-Govt. Lab., Akron 1, Ohio
Arthur F. Helin	Ditto
Leon E. Briggs	Naval Ord Lab, White Oak, Maryland
W. W. Rinne	Synthetic Rubber Div, RFC, Washington 25, D. C.
A. L. Hollis	Ditto
P. R. Stone	BuAer, Navy, Washington 25, D. C.
L. Boor	Phila QM Depot, Phila, Pa.
Philip Mitton	Engrs, Ft Belvoir, Va.
Clifford M. Brown	Ditto
D. M. French	Ditto
A. C. Hanson	Rock Island Arsenal, Rock Island, Illinois
R. F. Shaw	Ditto
R. M. Harper	Naval Air Exp. Station, Phila, Pa.
G. Reinsmith	ORDTB, OCO, Washington 25, D. C.

MINUTES OF
CONFERENCE ON LOW TEMPERATURE TESTING OF ELASTOMERS
4 and 5 March 1952

The printed record of the papers presented at this conference will be published with these minutes after approval by the agencies concerned.

The types of test apparatus used by each of the military laboratories were presented and discussed.

The following low temperature apparatus was selected for futher discussion which would lead to a choice for specifications:

Brittleness:

Impact type (motor or solenoid) ASTM D746
Bent loop type (hand operated) ASTM D736
Bent loop type - dead weight loading
Falling ball - ZZ-R-601

Hardness:

Shore A durometer)
Rex gage) variable hand load
Shore D durometer)
Pusey and Jones)
Admiralty meter) dead weight load
ASTM hardness)

Stiffness or flexibility:

Gehman torsional stiffness	ASTM D1053
Clash-Berg torsional stiffness	ASTM D1043
Werkenthin bending beam	
Young's Modulus	ASTM D797
Olson stiffness	ASTM D747
Compression-deflection	ASTM D575

Elastic recovery:

Compression set (Proposed ASTM D11 Section 17)
Temperature-retraction (Phillips and U.S. Rubber)
Tension recovery (Army Engineers and Army Quartermaster

Pressure sealing ASTM D1081

The conference agreed that the following test apparatus be standardized with a view towards limiting the use of other currently specified apparatus for specification purposes:

Brittleness:

Impact (Motor or Solenoid) ASTM D746 apparatus

Hardness:

Indentor - dead load type (ZZ-R-601 which is to include P & J and Admiralty)
Shore A durometer - secondary standard

Stiffness:

Gehman Torsional stiffness

Elastic recovery:

Compression set
Temperature-retraction

It is to be noted that agreement was reached on the type of test apparatus only; there was no agreement on test procedures, conditioning times or standard test temperatures. These latter items vary with the individual military services and their particular end item or service test requirements.

It was agreed to request Mr. Tener of the National Bureau of Standards to amend ZZ-R-601 now under revision to add or delete test methods as given in the following list:

Test Method	Action
5501 - Flexibility, bending beam	To remain
5502 - Brittleness, bent loop	To remain
5503 - Brittle point in liquids)	To be deleted and replaced by apparatus of ASTM D746
5504 - Brittle point in air)	
5505 - Brittle point, tubing	To be deleted
5506 - Contraction, linear	To be deleted
5507 - Flexibility, hose, hydrostatic	To remain
5508 - Compression set	To remain
5509 - Hardness, durometer	To remain
5510 - Hardness, plastometer	To remain
Gehman Torsional stiffness	To be added
Temperature-Retraction	To be added
Tension recovery	To be added

The conference members agreed to have a conference called by Army Ordnance in a year's time for the purpose of following up and implementing the decisions of this conference. A proposal for a round robin testing program will be considered at that time for the purpose of standardizing procedures, conditioning times and test temperatures.

R. F. SHAW
Secretary

INTRODUCTORY REMARKS OF COLONEL A.R. DEL CAMPO

I would like to take this opportunity to welcome you in behalf of Army Ordnance. The problem of low temperature testing of materials is an important one these days and any degree of improvement or standardization that can be achieved among the Services, as a result of conferences such as this, will be helpful, not only to the Services but also to Industry which must supply us with suitable materials. In the final analysis, when we say in our elastomer specifications that the material must be suitable at minus 65°F, minus 80°F or minus 100°F, our present tests are so general that the limits of these tests have only an empirical value and bear little relationship to properties expected under actual service conditions. I hope that you people will correct this condition by giving the Services methods and test equipment which, when used in specifications, will assure us procurement of the kind of rubber needed for good low temperature service.

STATUS OF LOW TEMPERATURE TEST METHODS IN PROPOSED REVISION OF FEDERAL SPECIFICATION 22-R-601 by R.F. TENER NATIONAL BUREAU OF STANDARDS
(Presented by Mr. I. Kahn)

Section 5000, Low Temperature Tests, of the proposed revision of Federal Specification ZZ-R-601a at this time describes the following 10 methods of test:

Flexibility, bending beam sometimes referred to as the Werkenthin cantilever beam apparatus -- or the Navy cantilever beam test.

Brittleness, bent loop, which is required in several Government specifications and is similar to A.S.T.M. D736-46T.

Brittle point in liquids. - This method is the old Bell Telephone method as modified, I believe, by the Navy Department. Specimen on a wheel-immersed in cold fluid 2 minutes - then strikes a bar.

Brittle point in air, in which the specimens are struck with a pendulum hammer.

Brittle point, tubing. - This is rather a specialized test and not too general in its applicability. The specimen is conditioned in a liquid bath and tested for brittleness on a pendulum type impact machine.

Flexibility, hose, hydrostatic, which is another test of limited applicability. Hoses are conditioned and filled with fluid - then flexed at the low temperature through 180°.

Contraction, low temperature linear, as developed by the Navy.

Compression set.

Durometer hardness.

Plastometer hardness.

These methods have been submitted to both Industry and the Government Departments for comment. The comments received from Industry were not very flattering and were not too constructive. Industry suggested that these methods be brought more in line with those of ASTM.

It was the consensus of opinion of Industry that the bent loop method should be deleted either because ASTM had dropped the method or that the bending beam method and the brittle point procedures would furnish sufficient information concerning the compound undergoing test. In addition, it was also pointed out in some comments that the bending beam and brittle point procedures are not as susceptible to personal errors as the bent loop. It is to be noted that the bent loop method, which was dropped by ASTM, has been proposed for reinstatement. The bending beam method, comparatively speaking, drew favorable comment.

The last three methods, compression set, durometer hardness, and plastometer hardness were approved as written. One reviewer stated that approval was not given on the basis of the information which the methods furnished but because consumers desired such tests; therefore, Industry would humor the consumer.

Comments from the Government Departments were in general more favorable than those from Industry, and were largely confined to clarification of the procedure. However, it was the general opinion that the bent loop method should be deleted and that some additional methods should be added,

such as: A torsion stiffness method such as Gehman, and brittleness test using the Brittleness Tester for Elastomers designed by the American Cyanamid Company.

However, due to the number and types of tests proposed, and the lack of evaluation of these procedures, the Committee has had to make a choice between methods of test that are now in use in Government specifications, and, including all methods that have been proposed irrespective of their present or prospective future use.

The latter procedure would have increased the number of tests included two or three times and with about the same number of dissatisfied customers. Certainly other methods will be added and methods now included will be dropped when available information indicates such a procedure is necessary or desirable.

It is hoped that agreement among Government agencies with respect to the relative value of the various low temperature tests will result from this meeting and that future cooperation of the group will be reflected in the procedures ultimately included in ZZ-R-601. Until this is accomplished, we may expect criticism of our lower temperature tests by non-Government groups to continue as in the last few years.

NOTE: This draft dated 29 March 1950, prepared by the Rubber Products Committee, contains section 5000 only. (See previous proposed documents (1) draft dated 23 September 1949 for preceding sections 1, 2, 3, 4, 5, 6, and 1000; (2) draft dated 27 December 1949 covering section 2000; (3) draft dated 21 February 1950 covering section 4000; and (4) draft dated 10 March 1950 covering, section 8000.) Remaining sections will follow at later dates. This draft has not been approved and is subject to modification. Comment Request No. 1304.

PROPOSED REVISION OF FEDERAL SPECIFICATION ZZ-R-601a

RUBBER GOODS; METHODS OF SAMPLING AND TESTING

(Section 5000)

This specification is a part of Section IV, Part 5, of the Federal Standard Stock Catalog.

SECTION 5000

THERMAL TESTS

I. High temperature

Method 5001 - Flame propagation

II. Low temperature

Method 5501 - Flexibility, bending beam

Method 5502 - Brittleness, bent loop

Method 5503 - Brittle point in liquids

Method 5504 - Brittle point in air

Method 5505 - Brittle point, tubing

Method 5506 - Contraction, low temperature, linear

Method 5507 - Flexibility, hose, hydrostatic

Method 5508 - Compression set

Method 5509 - Hardness, durometer

Method 5510 - Hardness, plastometer

FLAME PROPAGATION

1. Scope

1.1 This method is intended for use in determining the resistance of rubber and rubber-like materials to flame propagation.

2. Specimen

2.1 The specimen shall be 1/4-inch thick, 1/2-inch wide, and of any convenient length.

3. Apparatus

3.1 Candle - A standard candle, type II, Class B of Federal Specification No. C-C-91.

3.2 Timing device - A stop watch or other timing device which will indicate the time in seconds.

3.3 Shield - The shield shall be constructed from sheet metal or other fire-resistant material and shall be 12 inches wide, 12 inches deep, and 30 inches high, and open at the top. It shall be so constructed as to provide a ventilating opening approximately 1-inch in height around the bottom and shall have a viewing window in one side of sufficient size and in such a position that the entire length of the specimen being tested can be observed. One side of the shield shall be hinged (or some other suitable form of construction used) so that the shield may be readily opened and closed to facilitate the mounting and ignition of the specimen, Due to breakage of the glass window, it may be necessary to use heat-resistant glass for the viewing window.

3.4 Clamp - A spring-type paper clamp for holding the specimen in position shall be provided. The clamp shall be

attached rigidly to the shield in such a manner that when the specimen is clamped therein it will be centered within the shield facing the viewing window.

4. Procedure

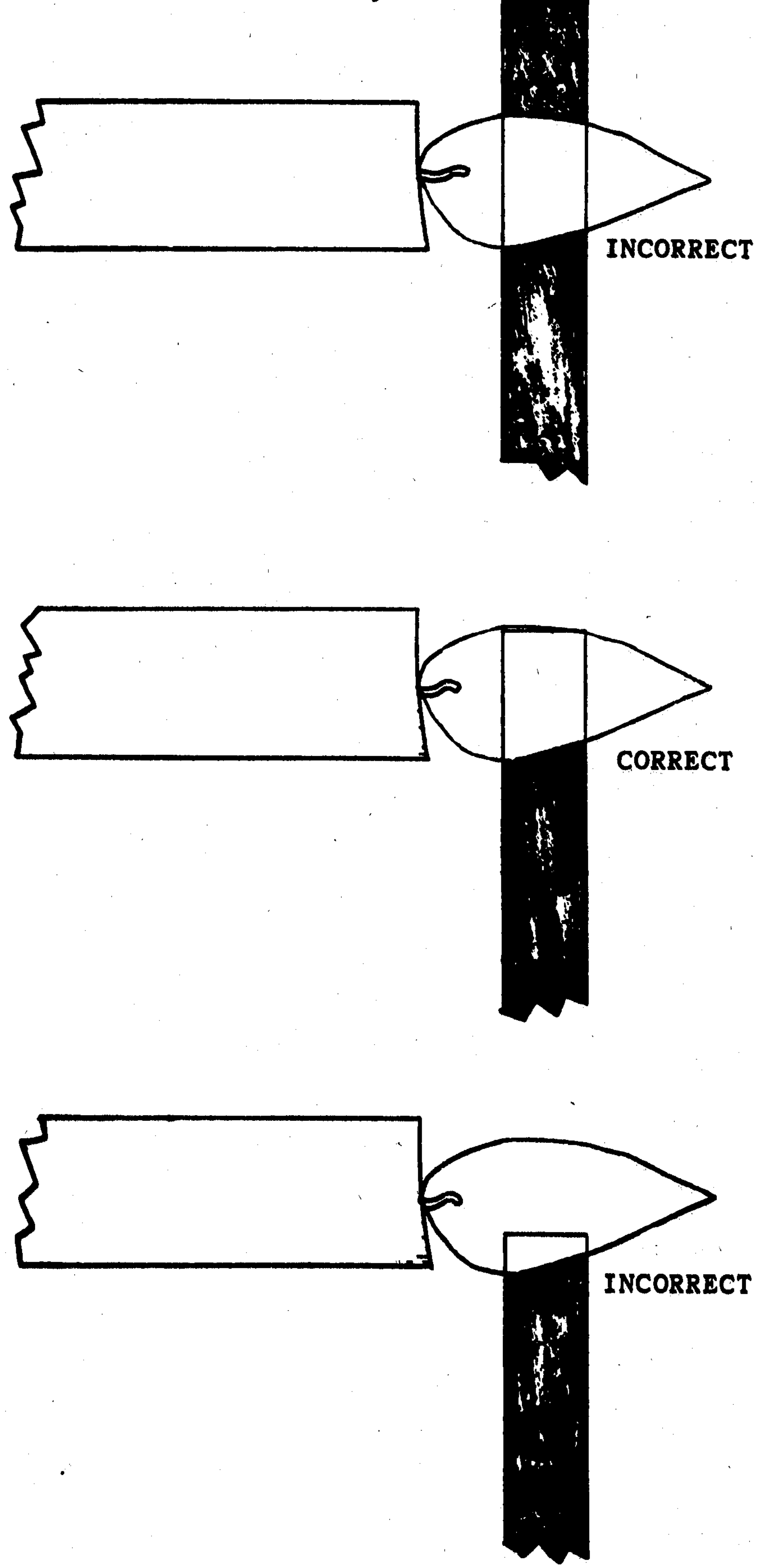
4.1 The specimen shall be lightly buffed (method 1002) and placed in the clamp in a horizontal position with the width direction of the specimen in a vertical position. The candle flame, protected from draft by the shield, shall be applied to the specimen in the manner shown in figure 5001 for exactly 1-minute. At the end of the 1-minute period, the flame shall be removed from the specimen and the time in seconds that the specimen continues to flame after removal shall be recorded as the flame propagation time.

5. Results

5.1 Unless otherwise specified in the detail specification, two specimens shall be tested. The average of the results obtained from the two specimens shall be the flame propagation time of the sample.

5.2 The flame propagation time shall be recorded to the nearest second.

FIGURE 5001



FLEXIBILITY-BENDING BEAM

1. Scope

1.1 This method is intended for use in determining the flexibility at low temperatures of rubber items such as gaskets and hose.

2. Specimen

2.1 The specimen shall be 10 inches long, 1-inch wide, and 1/4-inch thick and shall be taken from the finished item.

3. Apparatus

3.1 A low temperature cabinet of the type shown in figures 5501a and 5501c, which shall have a suitable control for maintaining the required temperature within plus or minus 2°F. The temperature shall be determined by a copper-constantan thermocouple and a potentiometer-type pyrometer or other equally accurate device.

3.2 A specimen rack in which the specimens can be clamped as shown in figure 5501b, shall be fitted in the cabinet. A string attached to the free end of the specimen shall come out through a hole in the cover of the box as shown in figure 5501a.

3.3 A spring scale graduated in grams (fig. 5501b) located outside the box and suspended directly over the specimen for measuring the load required to deflect the specimen. The string from the free end of the specimen shall be looped over the hook of the spring scale. The top of the spring scale shall be attached to either a manual or machine-driven pulley device in such a manner that the free end of the specimen will be raised at the rate of 1-inch per minute.

3.4 A measuring scale graduated in 1/32 of an inch for measuring the distance the specimen is deflected.

4. <u>Procedure</u>

4.1 The temperature of exposure of the specimen shall be as specified in the detail specification.

4.2 Unless otherwise specified in the detail specification, the specimen shall be exposed for 3 hours ± 1/4 hour before bending.

4.3 The specimen shall be clamped in position in the test rack so that the distance from the edge of the clamp to the point of attachment of the string to the specimen shall be 8 inches ± 1/32 inch. The rack with the specimens in position shall be placed in the cabinet and exposed for the required time at the required temperature.

4.4 At the end of the exposure period, an upward force just sufficient to take up the slack in the string shall be applied to the specimen through the spring scale, string, and pulley. Sufficient force to deflect the specimen at a rate of 1-inch per minute shall be applied and the force required to deflect the specimen 1-inch and 2 inches shall be read from the spring scale.

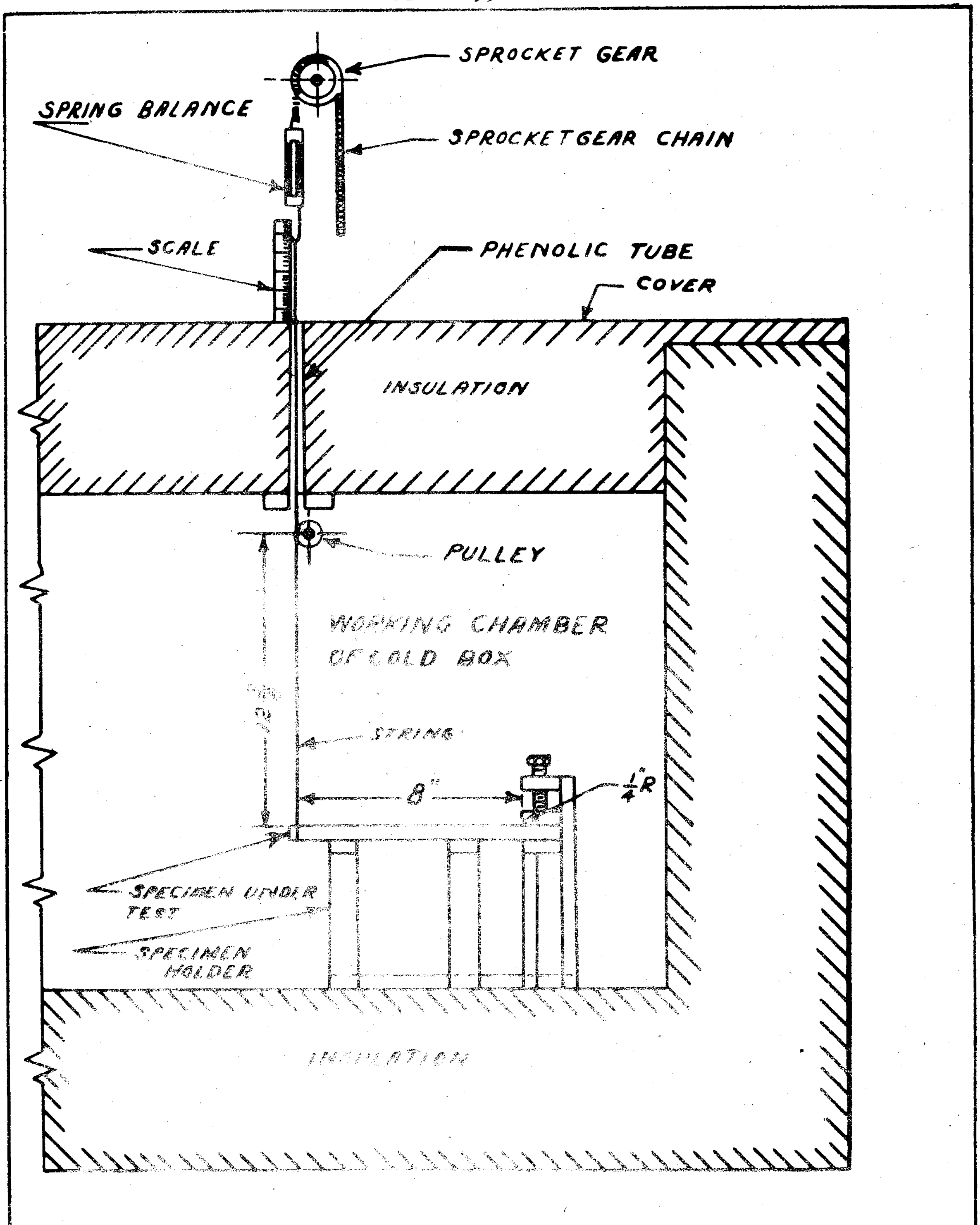
5. <u>Results</u>

5.1 Unless otherwise specified in the detailed specification, three specimens shall be tested. The average of the results obtained from the three specimens shall be the flexibility of the sample.

5.2 The force required to deflect the specimen shall be recorded to the nearest gram.

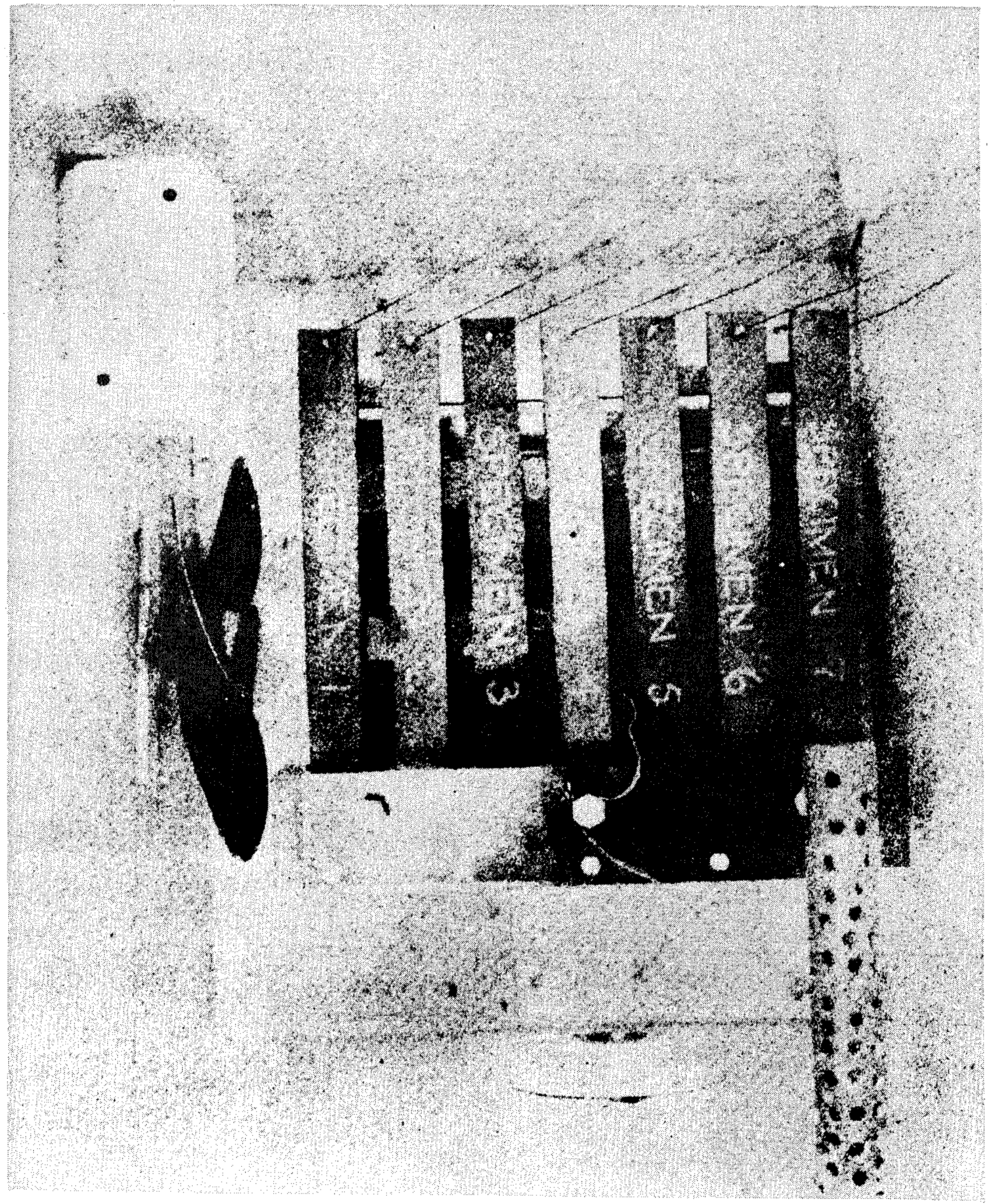
5.3 If the time of exposure is other than that required in 4.2, the time of exposure shall be recorded.

FIGURE 5501a



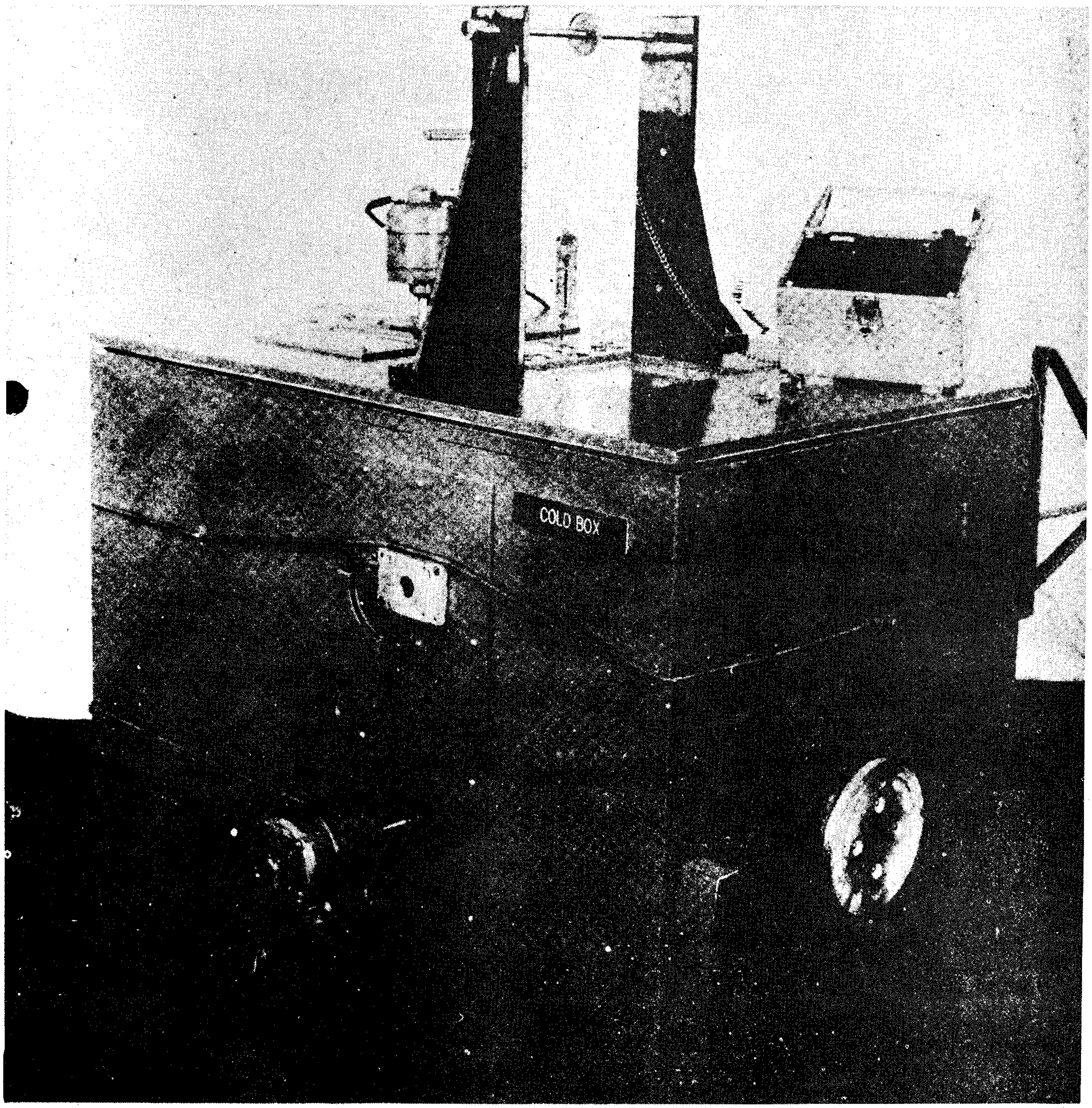
SCHEMATIC DIAGRAM SHOWING ESSENTIAL DIMENSIONS
OF SET-UP

FIGURE 5501b



INTERIOR VIEW OF COLD BOX SHOWING SPECIMENS IN POSITION FOR TEST.

FIGURE 5501c



VIEW OF COLD BOX SHOWING MECHANISM FOR APPLYING LOAD TO SPECIMENS.

BRITTLENESS, BENT LOOP

1. Scope

1.1 This method is intended for use in determining the ability of compounds made of rubber or rubber-like materials to resist the effect of low temperature in causing them to become brittle enough to fracture or crack when bent.

2. Specimen

2.1 The specimen shall conform in shape and dimensions to die II in method 2002.

3. Apparatus

3.1 The flexing fixture shall consist of two parallel plates each having a width of not less than 2 inches and so supported in guides that they may be rapidly moved from a position 2-1/2 inches apart until they are separated by a distance of 1-inch. Suitable clamping bars or devices shall be provided for holding the ends of the specimen for a distance of 1/4-inch at the corresponding edge of each plate so that when mounted, the specimen forms similar bent loops between the plates. A satisfactory flexing fixture is shown in figure 5502.

3.2 A low-temperature cabinet in which the specimen is exposed to the low temperature shall be of sufficient size to contain the flexing fixture unloaded with specimens, and so arranged as to permit the operation of the fixture to bend the specimens without removal from the chamber. The cabinet shall have a suitable control for maintaining within it the required temperature within plus or minus 3°F. The temperature shall be determined by a copper-constantan thermocouple and a potentiometer-type pyro-

meter or equally accurate device.

4. <u>Procedure</u>

4.1 The temperature of exposure shall be minus 40°F. or minus 70°F., as specified in the detail specification.

4.2 Unless otherwise specified in the detail specification, the time of exposure shall be 5 hours $\pm$ 1/4 hour.

4.3 The specimens shall be mounted in a looped position between the plates of the flexing fixture and shall be spaced at least 1/8-inch apart and held in the clamps for a distance of 1/4-inch from the end. With the plates of the flexing fixture in the open position, separated 2-1/2 inches, the fixture containing the specimens shall be placed in the cold chamber and exposed for the required period of time at the required temperature.

4.4 At the end of the exposure period and while still in the cold chamber, the plates of the flexing fixture shall be moved as rapidly as possible from the 2-1/2-inch distance of separation to a position where they are 1-inch apart. The specimen shall then be examined for fracture or visible cracks.

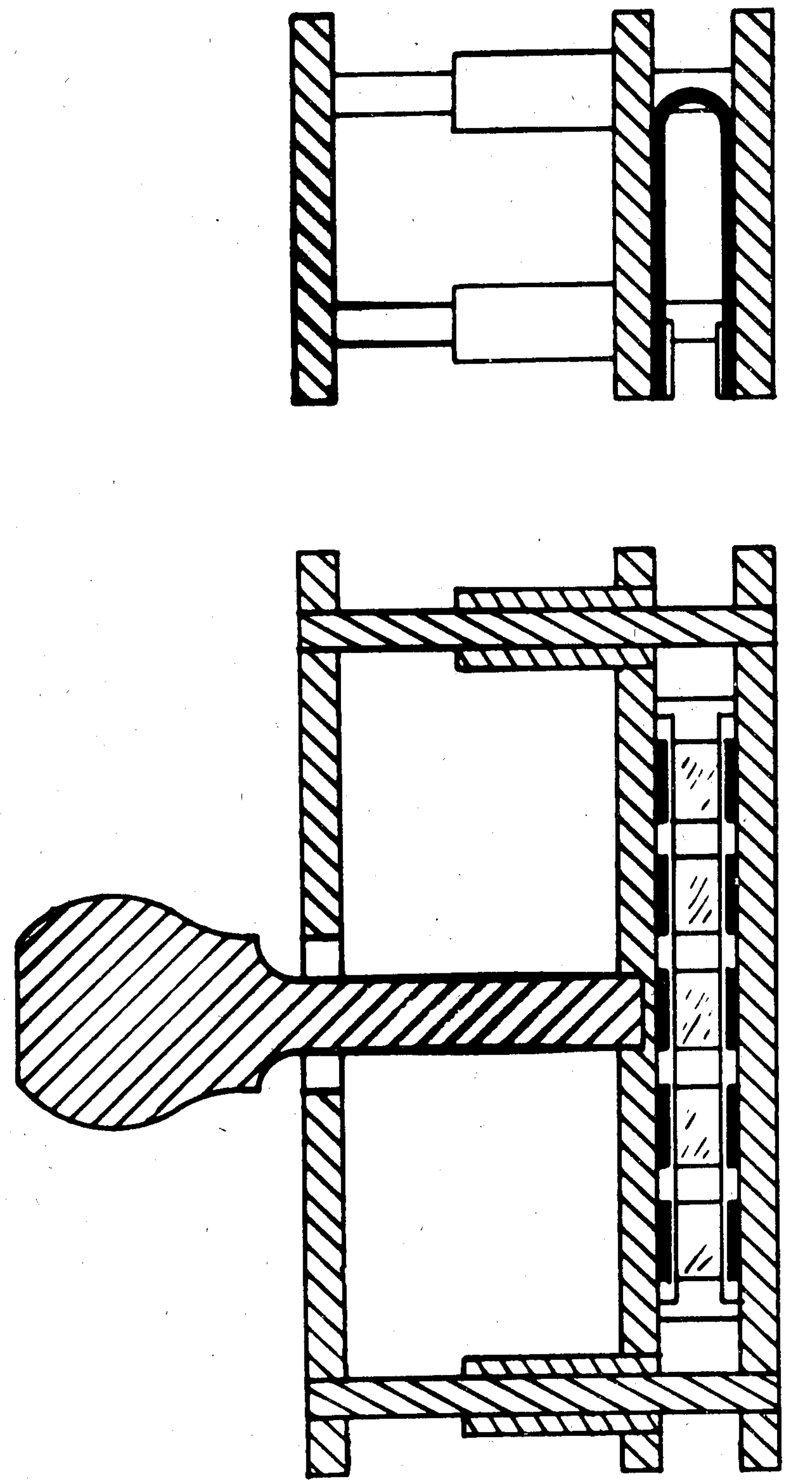
5. <u>Results</u>

5.1 Unless otherwise specified in the detailed specification, two specimens shall be tested.

5.2 The number of specimens which fracture or crack shall be recorded.

5.3 If the time of exposure is other than that required in 4.2, the time of exposure shall be recorded.

FIGURE 5502



BRITTLE POINT IN LIQUIDS

1. Scope

1.1 This method is intended for use in determining the brittle point of rubber compounds. The temperature at which a material breaks under a bending stress depends upon the thickness of the material and the extent and rate of bending. When these three variables are specified, the temperature at which a material breaks is termed its brittle point. This method employs a liquid cooling medium for use when speed is required.

2. Specimen

2.1 The specimen shall be 1-inch by 2 inches, cut by means of a die. Specimens over 0.085-inch in thickness shall be buffed (method 1002) on both sides to a thickness of 0.080 ± 0.005-inch.

3. Apparatus

3.1 A die for cutting the specimen.

3.2 An insulated steel tank, with inside dimensions of 18 by 2 by 8 inches, equipped with a stirrer. A brass semicircle with a 4.87-inch radius and a 0.50-inch thickness, for holding the specimen, shall be keyed to a shaft on the top of the tank. This shaft shall be located 7 inches from one end of the tank. A notch 0.25-inch deep and 0.075-inch wide shall be put in the rim of the semicircle. The notch shall be backed with a block having a 1/4-inch radius on the edge in contact with the specimen which shall extend 0.25-inch above the rim. A stiff, 1/4-inch round rod shall be fixed across the tank exactly 0.5-inch from the rim of the semicircle. The shaft upon which the sample-holding fixture is

mounted has a crank which, on rotation, shall immerse the specimen in the tank. The shaft shall be supported on ball bearings (figs. 5503a and 5503b).

3.3 A thermocouple for measuring the temperature of the cooling medium shall be located near the propeller of the stirrer

3.4 A close-fitting sheet metal hood shall be placed over the tank after inserting the specimen, to prevent the splashing of the cooling medium from the tank by rapid turning of the crank.

3.5 A liquid medium of low viscosity, even at the brittle point of the material under test, which shall not be a solvent or swelling agent for the material undergoing test.

3.6 Dry ice.

4. <u>Procedure</u>

4.1 The liquid cooling medium shall be as specified in the detail specification.

4.2 One end of the specimen shall be inserted in the notch on the rim of the semicircle. This can be done by grasping the corners of the specimen with pliers and stretching the rubber so as to decrease its thickness, allowing the specimen to slip freely into the notch. When the tension is relaxed, the specimen forms a snug fit in the notch and overlaps 1/4-inch on each side of the semicircle. Only one specimen at a time shall be inserted and tested on the rim of the semicircle in order to obtain maximum velocity of impact.

4.3 The tank shall be filled to within 3 inches of the top with the required cooling medium, which shall be

cooled to the desired temperature by means of small pieces of dry ice. The cooling medium shall be circulated by means of the stirring device. When the cooling medium arrives at the desired temperature, the semicircle shall be turned so that the specimen is immersed. Exactly 2 minutes after the specimen has been immersed, the crank shall be turned rapidly by hand in a clockwise direction. This causes the specimen to strike the horizontal bar just as the specimen rises out of the cooling medium. The temperature of the medium at which the specimen cracks shall be determined from the thermocouple and shall be considered to be the brittle point of the material.

5. Results

5.1 Unless otherwise specified in the detail specification, three specimens shall be tested. The average of the results obtained from the three specimens shall be the brittle point of the specimen.

5.2 The brittle point shall be recorded to the nearest 1°F.

5.3 Brittle point of different specimens of the same stock may be expected in general to agree within 0.5°F.

FIGURE 5503a

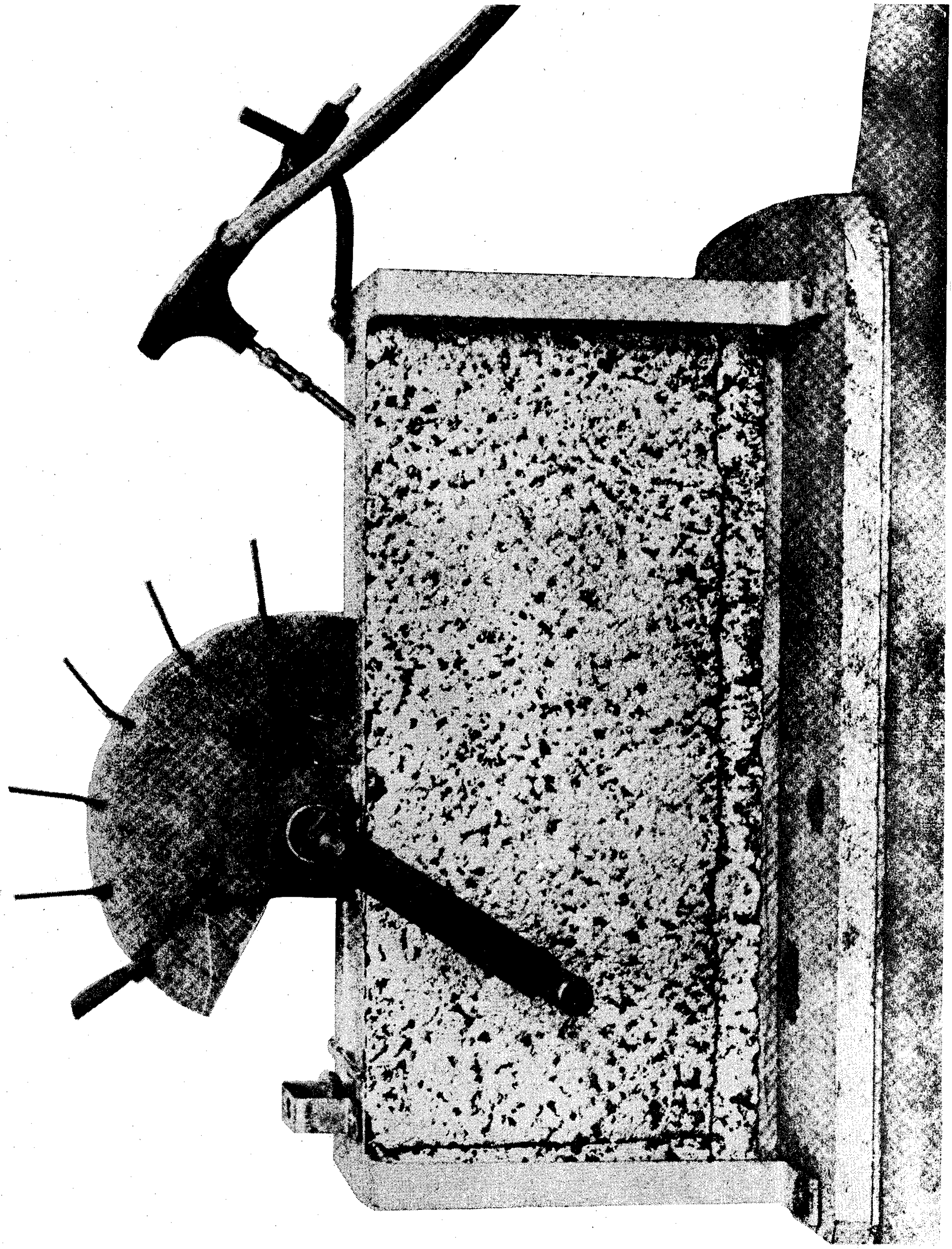
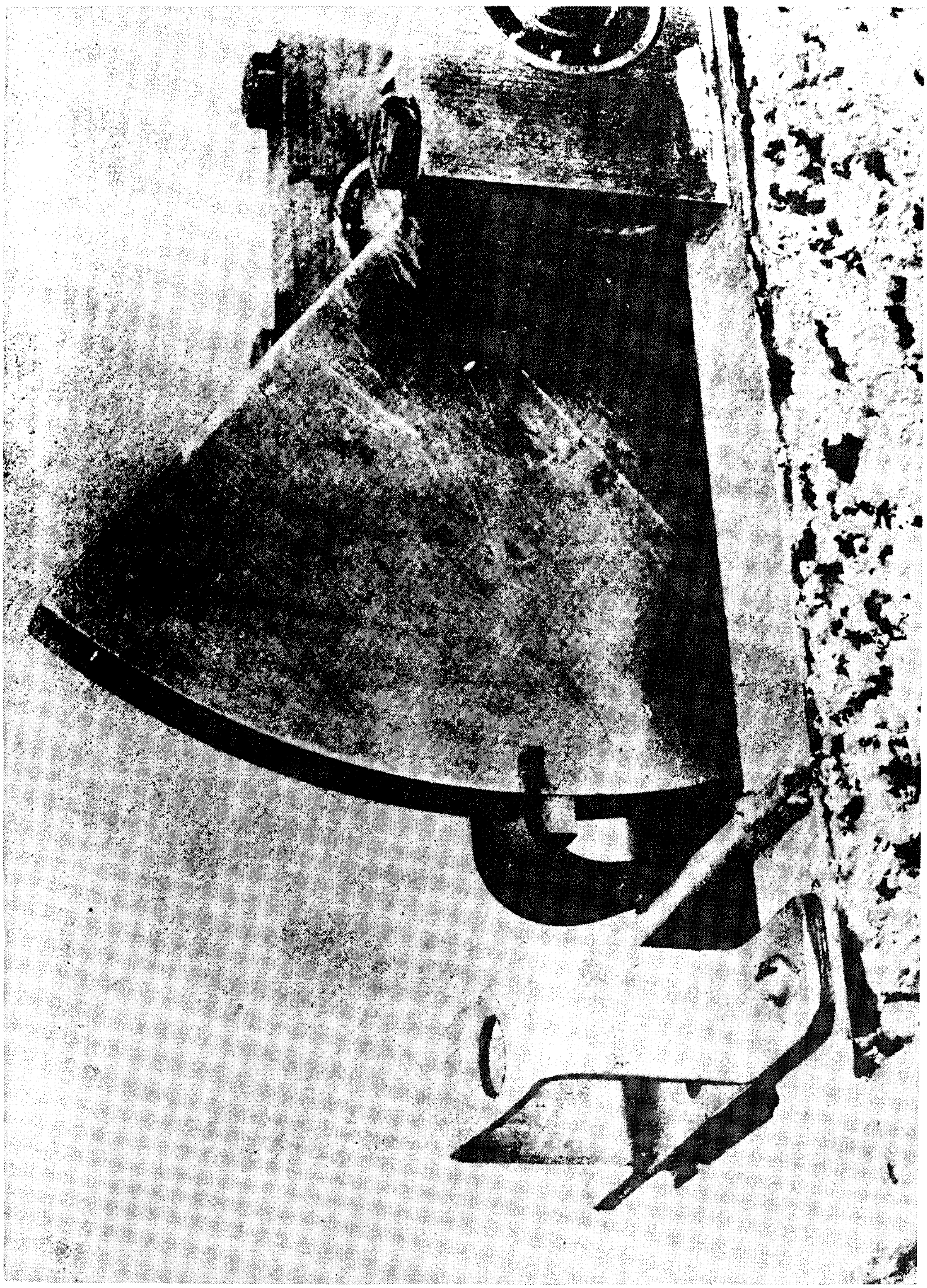


FIGURE 5503b



BRITTLE POINT IN AIR

1. Scope

1.1 This method is intended for use in determining the brittle point of a rubber compound where there is an objection to the method described in 5503 in that the specimens may be subject to extraction and/or swelling by the solvent used as the cooling medium.

2. Specimen

2.1 The specimen shall be 1-inch by 2-1/2 inches, cut by means of a die. Specimens over 0.085-inch in thickness shall be buffed (method 1002) on both sides to a thickness of 0.080 $\pm$ 0.005-inch.

3. Apparatus

3.1 A die for cutting the specimen.

3.2 A low-temperature cabinet with a suitable temperature control for varying the temperature of the working chamber in steps of 1°F.

3.3 A copper-constantan or other suitable thermocouple and potentiometer type pyrometer or other equipment of equal accuracy for measuring the temperature of the working chamber.

3.4 A specimen jig and a steel pendulum hammer for striking the specimen as shown in figure 5504a. The steel pendulum hammer shall be 3 inches long, 3 inches high, and 2 inches wide. The leading and following edge shall be rounded to a radius of 1-inch. The specimen jig shall be keyed to a shaft attached to a control plate outside the cabinet as shown in figure 5504b. The radius of swing shall be 8.5 inches with a clearance adjusted to 0.25-inch between

the lower surface of the hammer and the flats on the jig.

3.4.1 The mechanism described in method 5503 may be used in place of that described in 3.4.

3.5 A suitable clamping arrangement to hold the specimens in position on the jig.

4. Procedure

4.1 The specimen shall be placed in the jig and clamped into position so that each specimen projects 1-1/4 inches beyond the flat surface of the steel plate. The jig shall be mounted in the cold box and the controls set to obtain the desired temperature. The approximate brittle point of the material shall be determined by subjecting the specimen to two blows of the pendulum hammer at temperatures decreased in steps of 5°F., by dropping the pendulum from a level position, after the specimen shall have been conditioned for 10 minutes at each temperature. Views of the set-up, with the pendulum hammer raised to the striking position and with the pendulum at the instant of impact, are shown in figures 5504b and 5504c, respectively.

4.2 The brittle point may be determined more accurately by repeating the test in steps of 1°F. decrease in temperature in the brittleness range of the material. The temperature at which the specimen breaks under a single blow of the hammer after being exposed for 10 minutes at that temperature shall be recorded at the brittle point of the specimen.

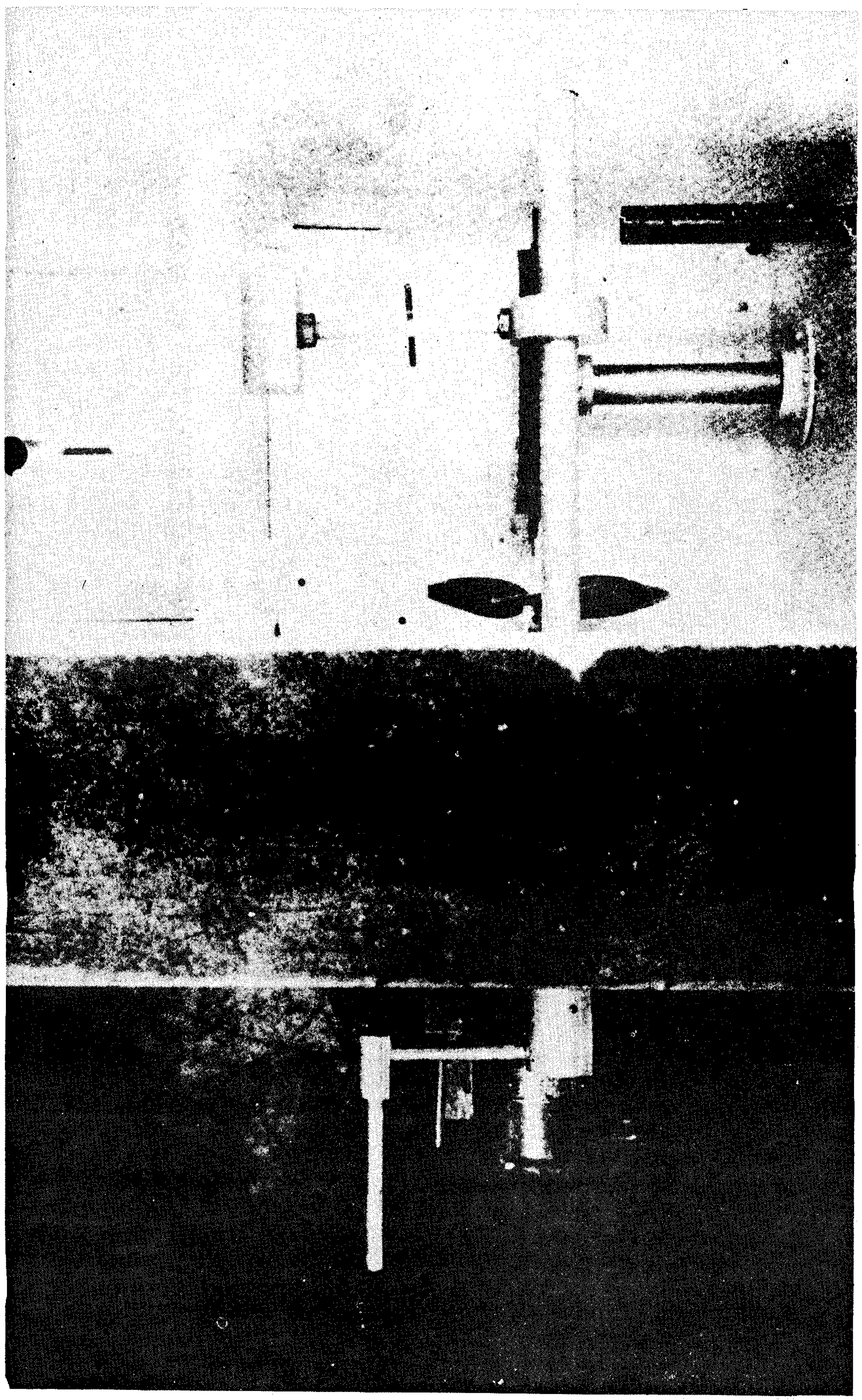
5. Results

5.1 Unless otherwise specified in the detail specification, three specimens shall be tested. The average of

the results obtained from the three specimens shall be the brittle point of the sample.

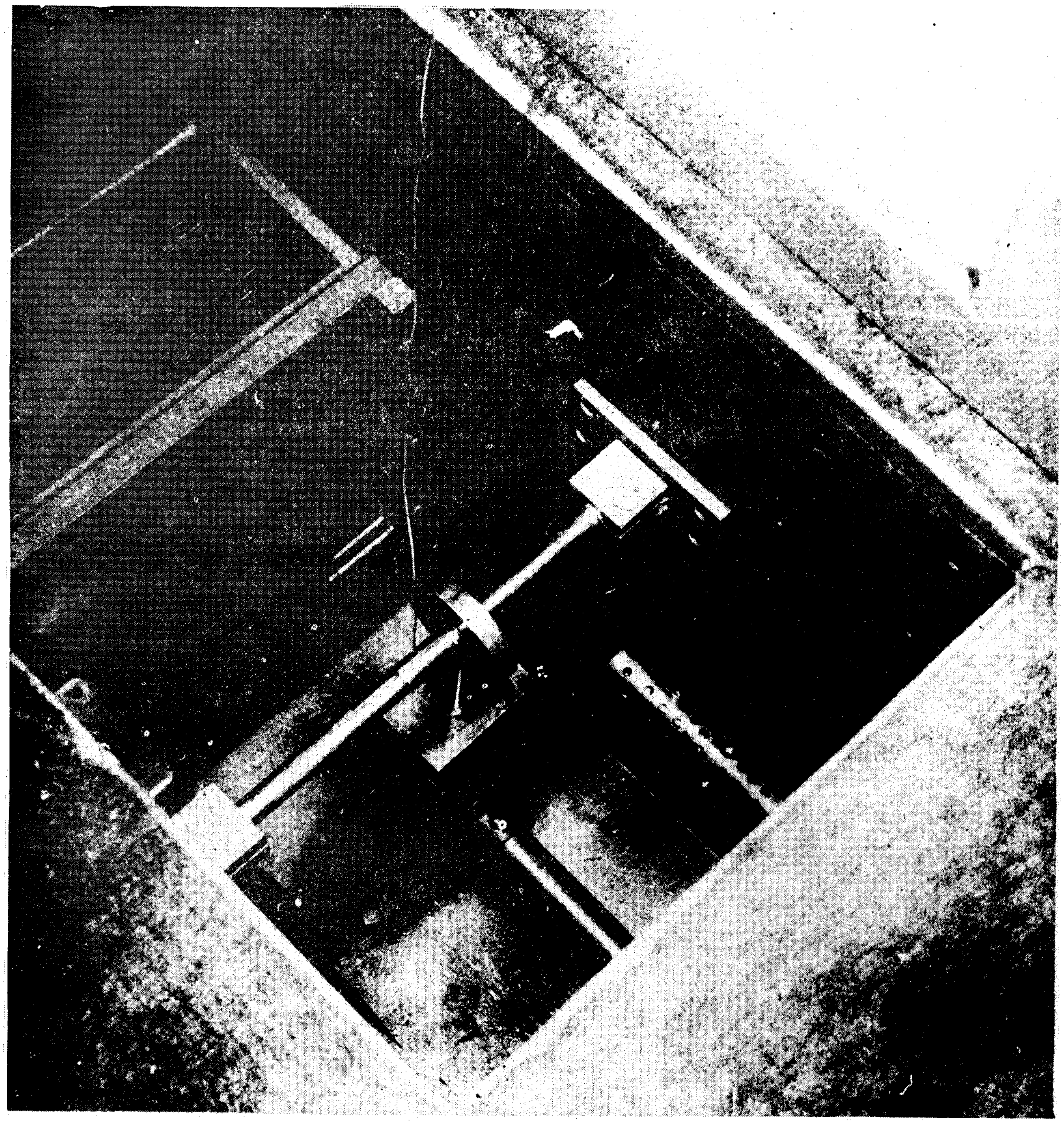
5.2 The brittle point shall be recorded to the nearest 5°F. or 1°F., depending upon whether the procedure in 4.1 and 4.2, respectively, is used.

FIGURE 5504b



HAMMER OF BRITTLE POINT APPARATUS POSITIONED FOR IMPACT BLOW

FIGURE 5504c



BRITTLE POINT TEST, TUBING

1. Scope

1.1 This method is intended for use in determining a temperature above which the tubing under test is not brittle.

2. Specimen

2.1 When the tubing has an outside diameter of less than 0.50-inch, the specimen shall consist of a 3-inch length. When the tubing has an outside diameter greater than 0.50-inch, the specimen shall consist of a 5-inch length.

2.2 Both ends of the specimen shall be closed with corks and a thermocouple shall be located in the center of the specimen as shown in figure 5505a.

3. Apparatus

3.1 An impact tester of the pendulum type equipped for the simple beam or Charpy impact test as shown in figures 5505b and 5505c.

3.2 The dimensions of the machine shall be such that the center of percussion of the striker is at the point of impact, that is, the center of the striking edge.

3.3 The pendulum shall be so constructed that when released from such a position that the linear velocity of the center of the striking edge (center of percussion) at the instant of impact shall be approximately 11 feet per second, which corresponds to an elevation of this point of 2 feet.

3.4 The striking edge of the pendulum shall be tapered to have an included angle of 45°and shall be rounded to a radius of 0.125-inch.

3.5 A liquid bath in which the temperature can be controlled to plus or minus 2°f.

3.6 A liquid medium of low viscosity which shall not affect the material undergoing test.

3.7 Dry ice for cooling the bath.

4. Procedure

4.1 The liquid cooling medium shall be as specified in the detail specification.

4.2 The specimen shall be supported against two rigid blocks in such a position that its center of gravity shall lie on a tangent to the arc of travel of the center of percussion of the pendulum drawn at the position of impact. The edges of the blocks shall be rounded to a radius of 0.125-inch. The points of support shall be 2 inches apart for specimens having an outside diameter less than 0.50-inch and 4 inches apart for specimens having an outside diameter greater than 0.50-inch.

4.3 The bath shall be brought to the required temperature with dry ice. The specimen shall be placed in the bath and maintained until the thermocouple on the interior of the specimen registers the same temperature as that of the bath. When the specimen has reached the required temperature, it shall be removed from the bath, placed on the supports as the impact tester as shown in figure 5505c and tested as quickly as possible. In breaking the specimen, the pendulum shall be released from such a position that the linear velocity of the center of the striking edge (center of percussion) at the instant of impact shall be approximately 11 feet per second, which corresponds to an elevation of this point of 2 feet. The temperature at which these specimens cracks or shatters regardless of the residual force of the pendulum shall be re-

corded as the brittle point.

5. Results

5.1 Unless otherwise specified in the detail specification, three specimens shall be tested. The average of the results obtained from the three specimens shall be the brittle point of the sample.

5.2 The brittle point shall be recorded to the nearest 2°F.

FIGURE 5505a

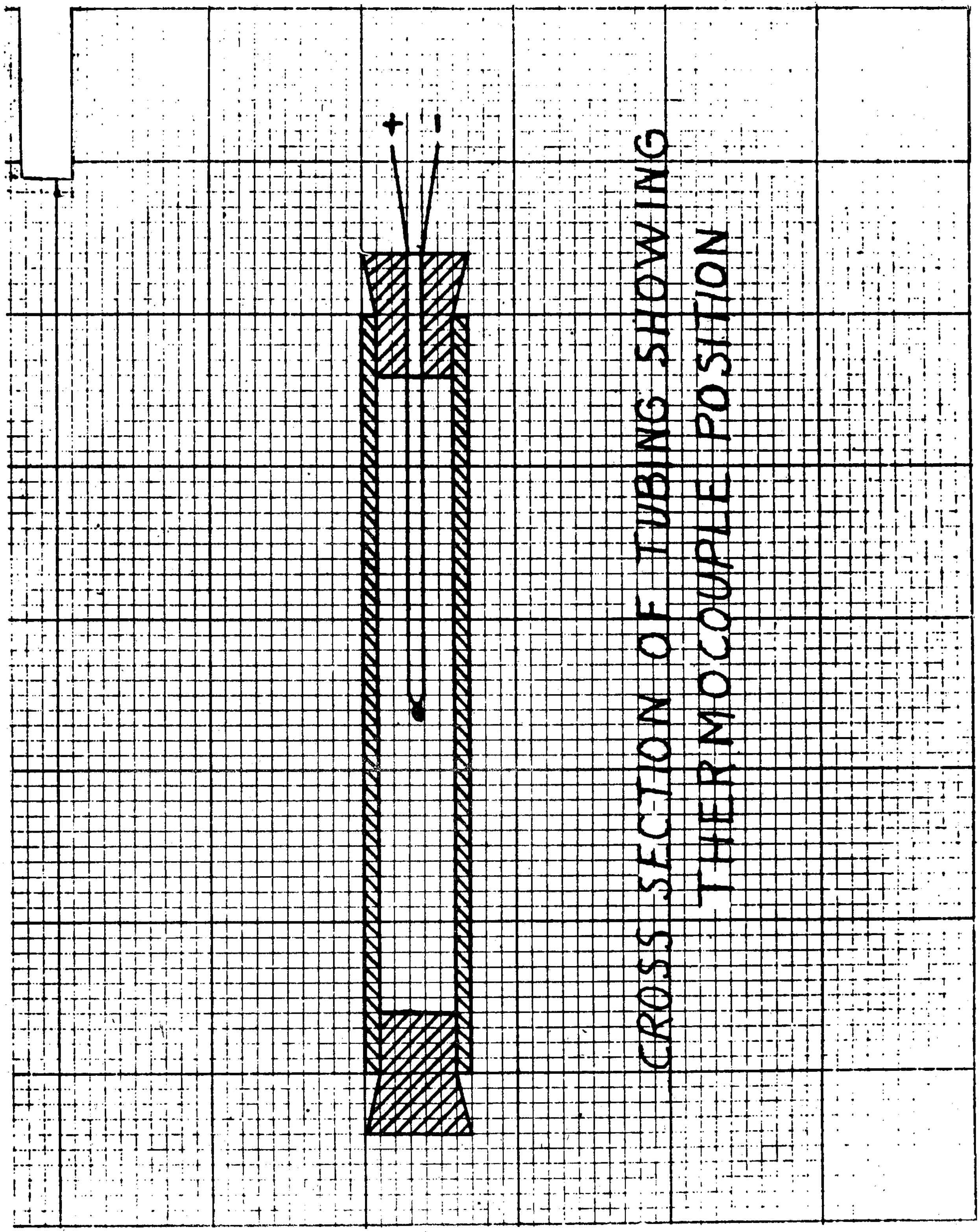


FIGURE 5505b

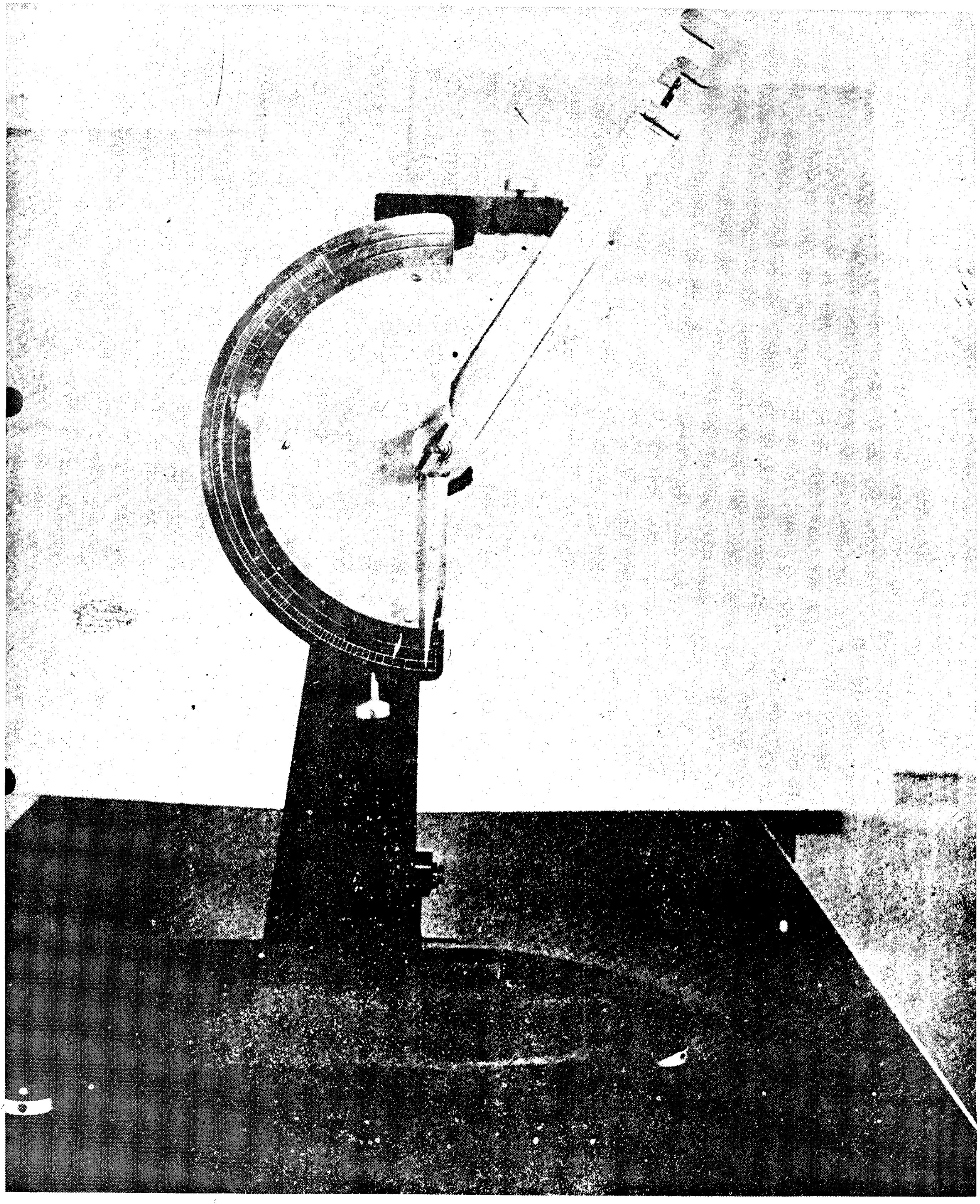
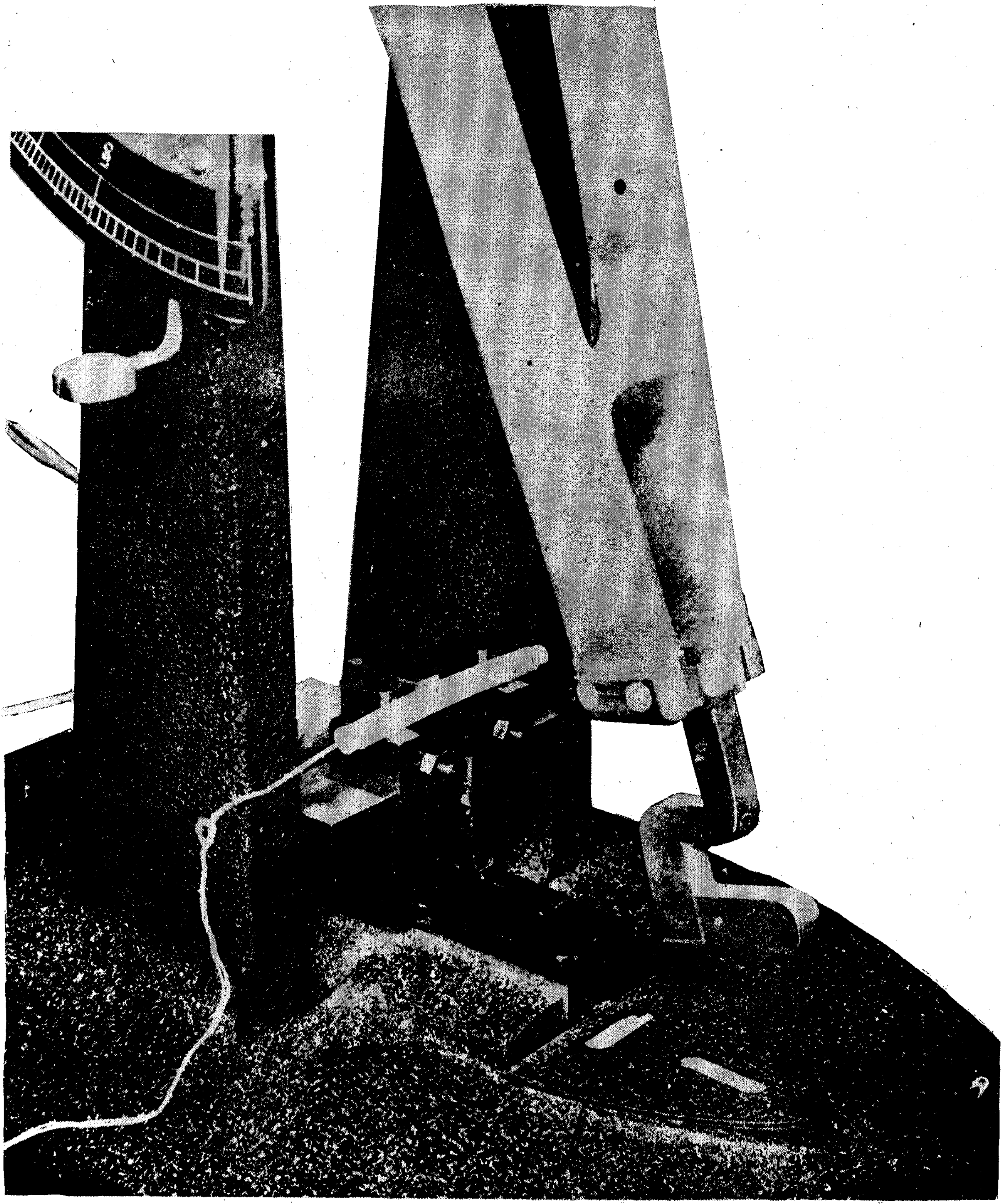


FIGURE 5505c



CONTRACTION, LOW TEMPERATURE,
LINEAR

1. <u>Scope</u>

1.1 This method is intended for use in determining the linear contraction of an elastomeric material when subjected to low temperatures.

2. <u>Specimen</u>

2.1 The specimen shall be a 6-inch square and shall be 0.07 to 0.08-inch in thickness.

3. <u>Apparatus</u>

3.1 A low temperature cabinet which shall have a suitable control for maintaining the required temperature within plus or minus 2°F. and large enough to maintain the rubber specimen and provide room for inscribing with the dividers.

3.2 A pair of sharp pointed dividers or equivalent instrument.

3.3 A wood support and assembly as shown in figure 5506.

3.4 A thermocouple or other equivalent apparatus for measuring the temperature of the cabinet.

4. <u>Procedure</u>

4.1 Unless otherwise specified in the detail specification, the temperature shall be - 65° ± 2°F.

4.2 The specimen shall be mounted on the wood board and a 4-inch radius, 90°-arc shall be scribed on the surface of the specimen at a temperature of 70° ± 5°F. The specimen shall then be subjected to the required temperature and the same radius scribed after 24 hours and again after 72 hours at this temperature.

4.3 The platen specimen shall be allowed to return

to room temperature ($70^\circ \pm 5^\circ$F.) and the distance measured between the original scribed mark and the two scribed marks made at the low temperature both with and against the grain.

4.4 The dividers shall be kept at room temperature at all times except when scribing the marks at low temperature.

5. Results

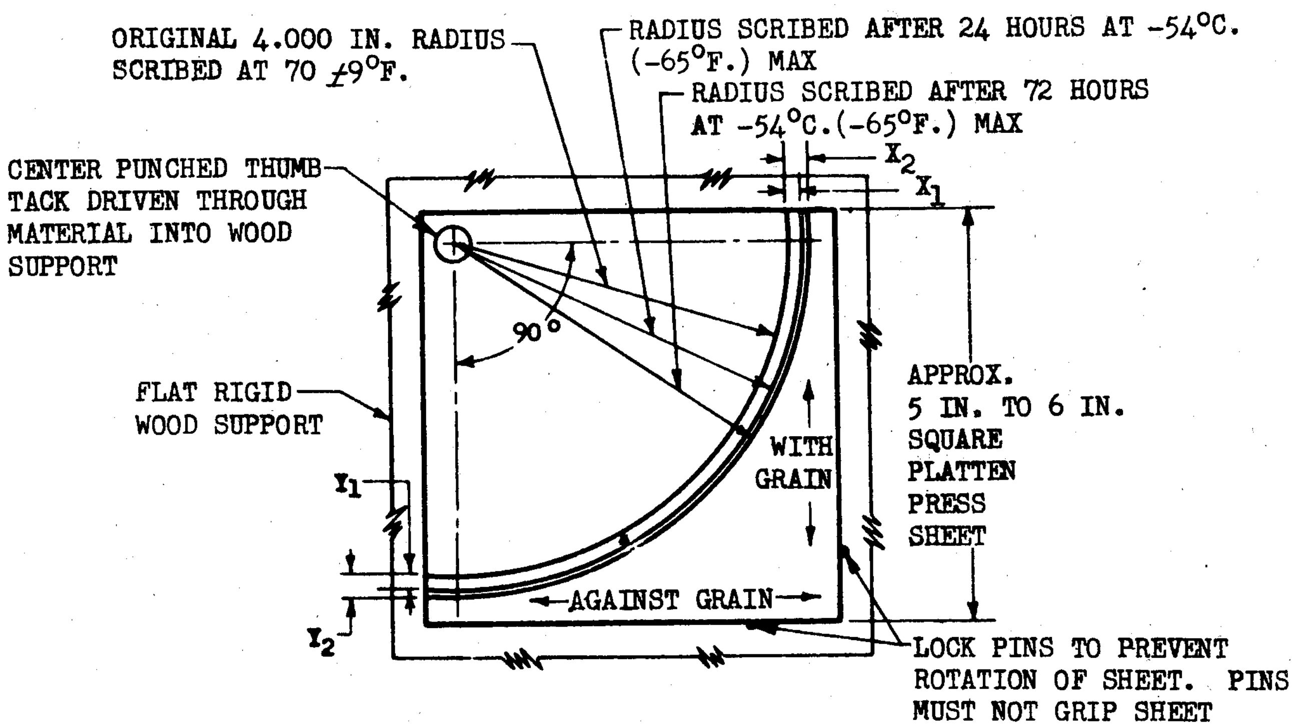
5.1 The contraction shall be calculated as follows:

$$\text{Contraction, percent} = \frac{100 \times \text{the difference between scribed marks}}{\text{radius}}$$

5.2 Unless otherwise specified in the detail specification, two specimens shall be tested. The average of the results obtained from the two specimens shall be the contraction of the sample.

5.3 The contraction both with and across the grain shall be recorded to the nearest 0.1 percent.

FIGURE 5506



MEASURE X AND Y INCREMENTS ACCURATELY ON 90° CENTER LINES AFTER MATERIAL HAS RETURNED TO 21 ±5°C. (70 ±9°F.).

SCRIBED ARCS MADE WITH SHARP DIVIDERS HELD TO SAME ADJUSTMENT AND 75 ±5°F. TEMPERATURE. DO NOT LEAVE DIVIDERS IN COLD BOX LONGER THAN NECESSARY TO SCRIBE ARCS. AREA TO BE SCRIBED MAY BE LIGHTLY BUFFED OR COVERED WITH CHALK.

Figure 3. - Linear construction test details.

FLEXIBILITY, HOSE, HYDROSTATIC

1. Scope

1.1 This method is intended for use in determining the flexibility of hose under hydrostatic pressure at low temperatures.

2. Specimen

2.1 The specimen shall consist of two lengths of hose, each 18 inches long.

3. Apparatus

3.1 A cold chamber equipped with a temperature control that will maintain the temperature of the chamber within plus or minus 2°F. of the required temperature for a period of at least 24 hours. The chamber shall be of sufficient size to permit flexing the specimen through an angle of 180°.

3.2 An immersion tank of sufficient size for immersing the specimen in a liquid and equipped with a temperature control that will maintain the required temperature within plus or minus 2°F for 7 days.

3.3 Hydraulic pressure equipment consisting of a hand pump, gage, release valve, shut-off valve, and accumulator.

3.4 The required immersion hydraulic fluid. (The fluid used should be that with which the hose is used in service.)

3.5 An air oven equipped with controls that will maintain the required temperature within plus or minus 2°F.

3.6 A satisfactory assembled apparatus is shown in figure 5507.

4. Procedure

4.1 Unless otherwise specified in the detail specifi-

cation, the temperature of exposure in the c old chamber shall be $-40 \pm 2^{\circ}F.$ and the time of exposure shall be at least 24 hours.

4.2 The immersion and hydraulic fluid shall be as specified in the detail specification.

4.3 Unless otherwise specified in the detail specification, the temperature of immersion of the hose shall be $70^{\circ} \pm 1^{\circ}C.$ ($158^{\circ} \pm 2^{\circ}F.$) and the time of immersion shall be 168 hours.

4.4 Unless otherwise specified in the detail specification, the temperature of exposure of the hose in air shall be $70^{\circ} \pm 1^{\circ}C.$ ($158^{\circ} \pm 2^{\circ}F.$) and the time of exposure shall be 168 hours.

4.5 One length of the hose shall be immersed in the immersion medium for the required time at the required temperature (see4.3). The other length of hose shall be exposed in air at the required temperature for the required time (see 4.4).

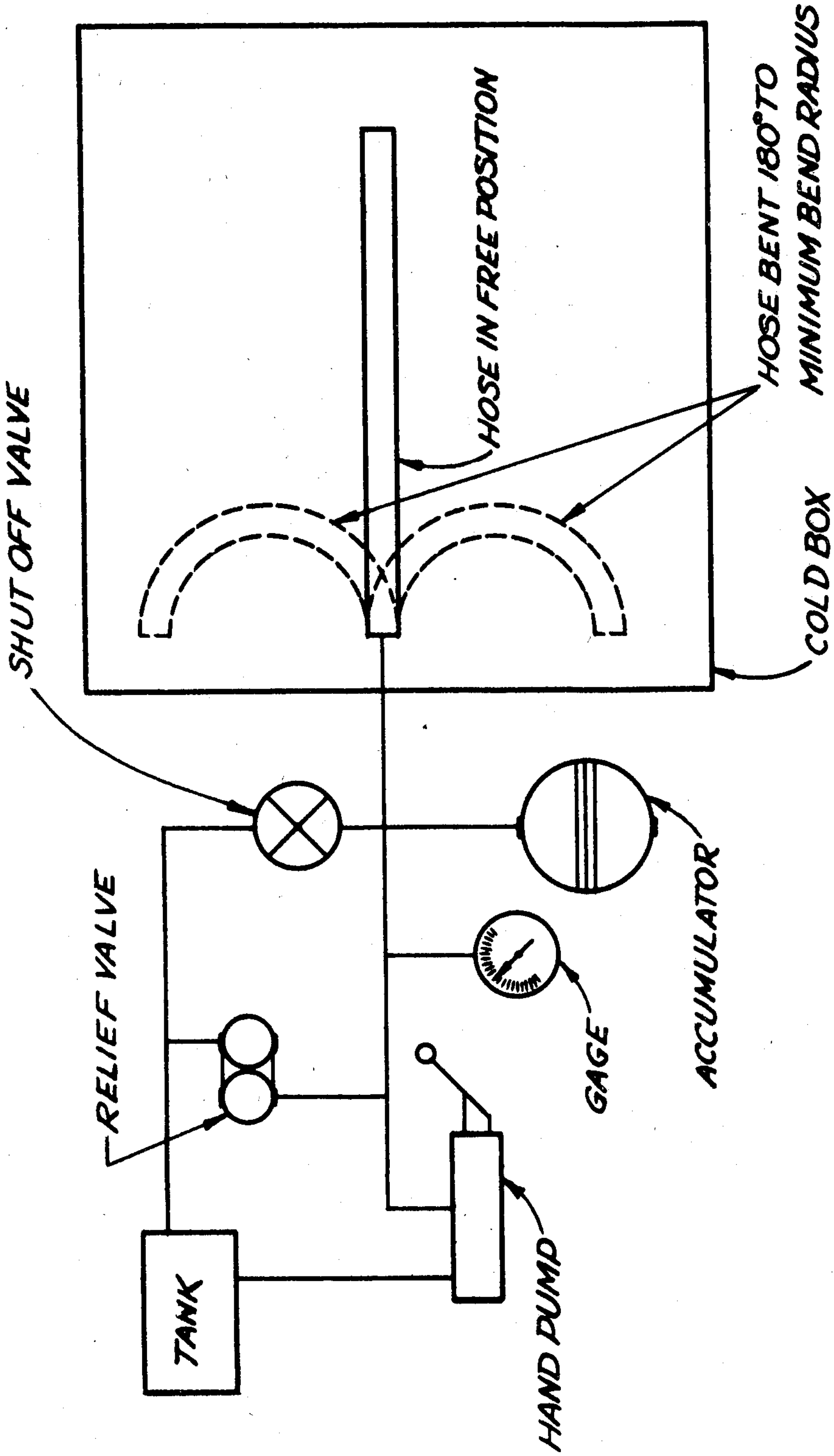
4.6 Both lengths of the specimen shall then be filled with the required hydraulic fluid and placed in the cold chamber at the required temperature for the required time (see 4.1). While still in the low-temperature chamber at the required temperature, a hydraulic pressure shall be applied to the lengths of hose equivalent to the maximum service pressure of the hose and the lengths shall be flexed through 180° to the minumum bend radius of each extreme of travel. The lengths shall be subjected to 5 cycles at a rate of 1 cycle in 4 seconds.

5. <u>Results</u>

5.1 Unless otherwise specified in the detail specification, two specimens shall be tested.

5.2 Any leakage, cracking, or failure of the specimen shall be recorded.

FIGURE 5507


HOSE BENT 180° TO
MINIMUM BEND RADIUS
HOSE IN FREE POSITION
SHUT OFF VALVE
COLD BOX
ACCUMULATOR
GAGE
RELIEF VALVE
TANK
HAND DUMP

COMPRESSION SET

1. Scope.

1.1 This method is intended for use in determining the compression set of rubber at low-temperature.

2. Specimen

2.1 The specimen shall be as described in method 3006.

3. Apparatus

3.1 A suitable container wherein liquid or air may be maintained at the required temperature within plus or minus 2°f.

3.2 Thickness gage as described in method 3006.

3.3 Compression set apparatus as described in method 3006.

3.4 Apparatus for preparing the specimen as described in method 3006.

3.5 Apparatus as described in method 5501 for measuring the temperature.

4. Procedure

4.1 The procedure shall be as described in method 3006 with the exception given below:

4.2 The specimen shall be placed in the compression set device at a temperature of 70° to 90°F.

4.3 The time and temperature of exposure shall be as specified in the detail specification.

4.4 When a liquid medium is used, it shall be as specified in the detail specification.

4.5 At the end of the exposure period, the compression on the specimen shall be released while in the low-temperature medium.

4.6 The thickness of the specimen after releasing the

compression shall be determined immediately and while the specimen is still in the low-temperature medium. The thickness gage shall be at the temperature of the specimen.

5. Results

5.1 The results shall be described in method 3006. In addition, if a liquid cooling medium is used, it shall be recorded.

HARDNESS, DUROMETER

1. Scope

1.1 This method is intended for use in determining the durometer hardness of rubber at a low temperature.

2. Specimen

2.1 The specimen shall be as described in method 3002.

3. Apparatus (See Fig. 5509.)

3.1 A durometer as described in method 3002.

3.2 A low-temperature cabinet which shall have a suitable control for maintaining the required temperature within plus or minus 2°F. during the exposure period.

3.3 Apparatus described in method 5501 for measuring the temperature.

3.4 Apparatus for applying the durometer to the specimen while at the temperature of test.

4. Procedure

4.1 The temperature of test and the time of exposure of the specimen shall be as specified in the detail specification.

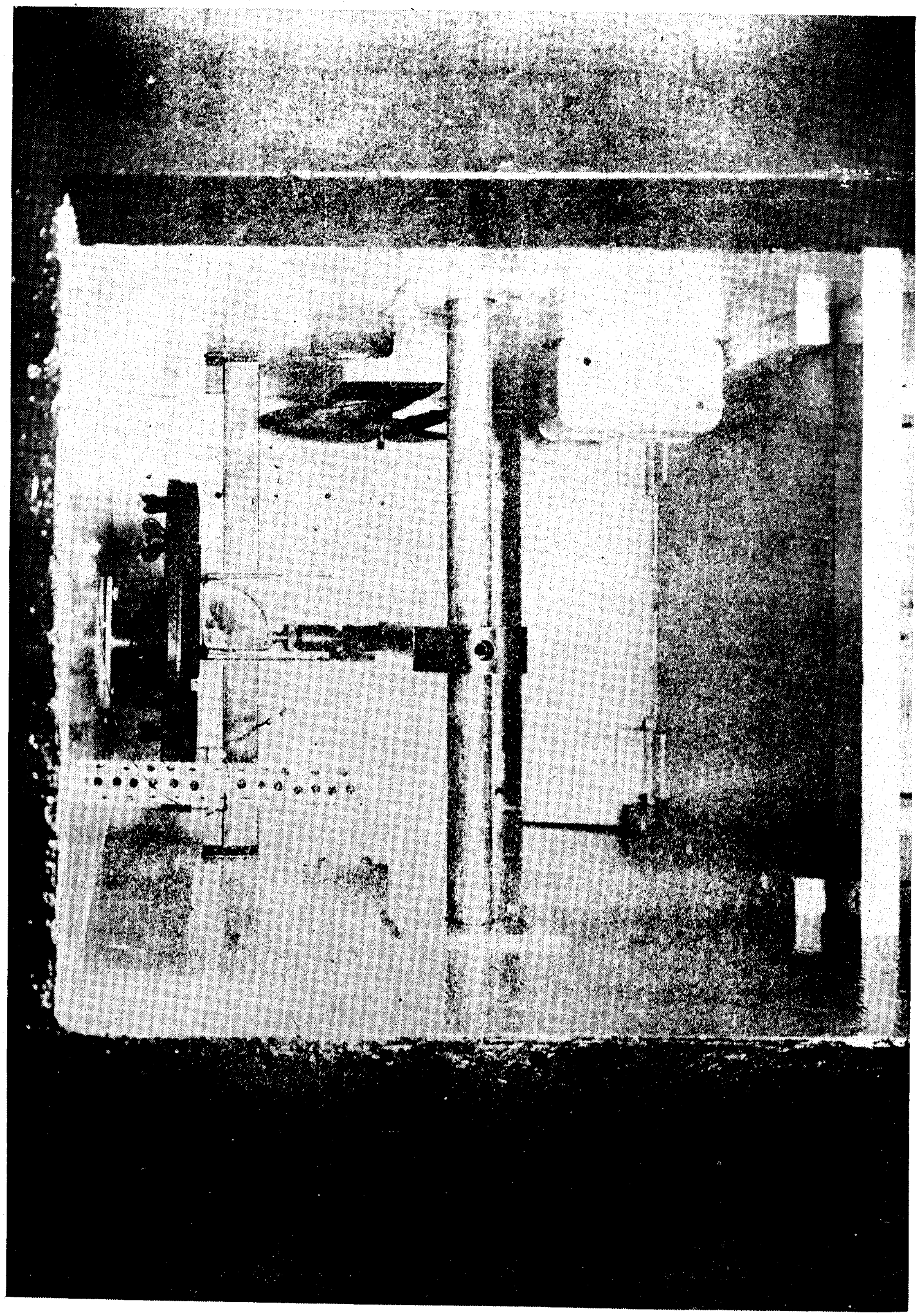
4.2 At the end of the exposure period, the hardness of the specimen shall be determined at the exposure temperature as described in method 3002 except that, unless otherwise specified in the detail specification, a pressure of 2 pounds shall be applied to the durometer during the test. The durometer shall be at the same temperature as the specimen.

5. Results

5.1 The results shall be as described in method 3002.

5.2 When the pressure on the durometer is different from that given in 4.2, it shall be recorded.

FIGURE 5509



HARDNESS, PLASTOMETER

1. Scope.

1.1 This method is intended for use in determining the plastometer hardness of rubber at low-temperature.

2. Specimen

2.1 The specimen shall be as described in method 3001.

3. Apparatus

3.1 A plastometer as described in method 3001.

3.2 A low-temperature cabinet or liquid bath which shall have a suitable control for maintaining the required temperature within plus or minus 2°F. during the exposure.

3.3 Apparatus as described in 5501 for measuring the temperature.

3.4 A leveling platform (fig. 5510a). The strips on top of the platform for maintaining the plastometer shall be made of any good insulating material such as phenol formaldehyde insulating material.

3.5 An extension of the same insulating material as used for the strips (see 3.4) for lengthening the indentor shaft of the plastometer (fig. 5510b). If an aluminum shaft is made to replace the original shaft in the plastometer, the insulator may be added without changing the force impressed on the specimen exclusive of the movable weight.

4. Procedure

4.1 The temperature of test and time of exposure shall be as specified in the detail specification.

4.2 The liquid media, when used, shall be as specified in the detail specification.

4.3 For moderately low-temperatures, the hardness of

the specimen shall be determined after the specimen and apparatus has been conditioned at the required temperature and for the required time as described in method 3001. The plastometer shall be at the same temperature as the specimen.

4.4 When extremely low-temperatures of exposure are required or when the apparatus cannot be operated satisfactorily in cold air because of operating difficulties, the following method shall be used:

4.4.1 A low-temperature liquid bath shall be adjusted to the required temperature. The leveling platform (see 3.3) shall be placed in the bath so that the surface of the liquid is flush with the top surfaces of the insulating strips and allowed to come to the temperature of the bath.

4.4.2 The specimen shall be placed in the leveling platform between the insulating strip, below the surface of the liquid, and conditioned for the required time.

4.4.3 The plastometer with the insulated shaft in position shall be placed on the platform as shown in figure 5510c and the hardness determined as described in method 3001.

4.4.4 The indentor point shall be at the same temperature as the specimen.

5. Results

5.1 The results shall be as described in method 3001.

FIGURE 5510a

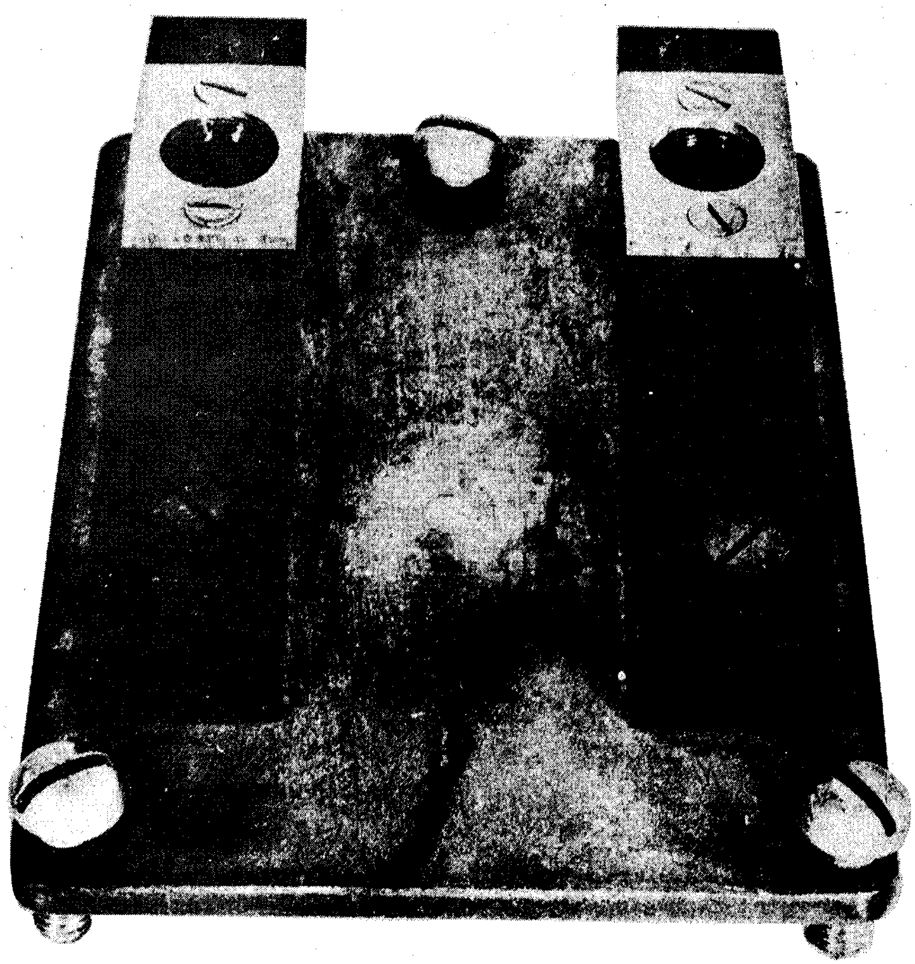


FIGURE 5510b

FIGURE 5510c

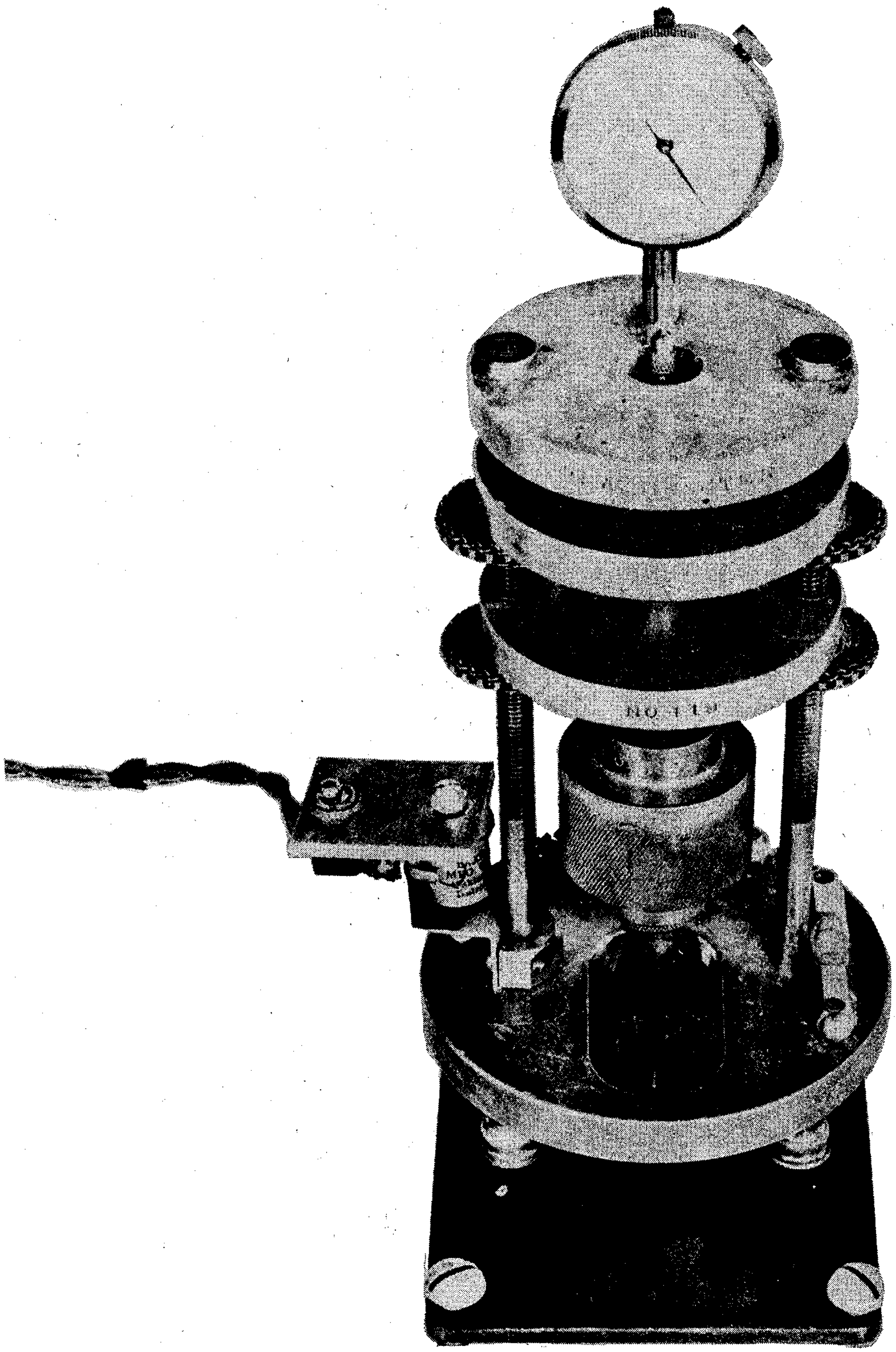


FIGURE 5513b

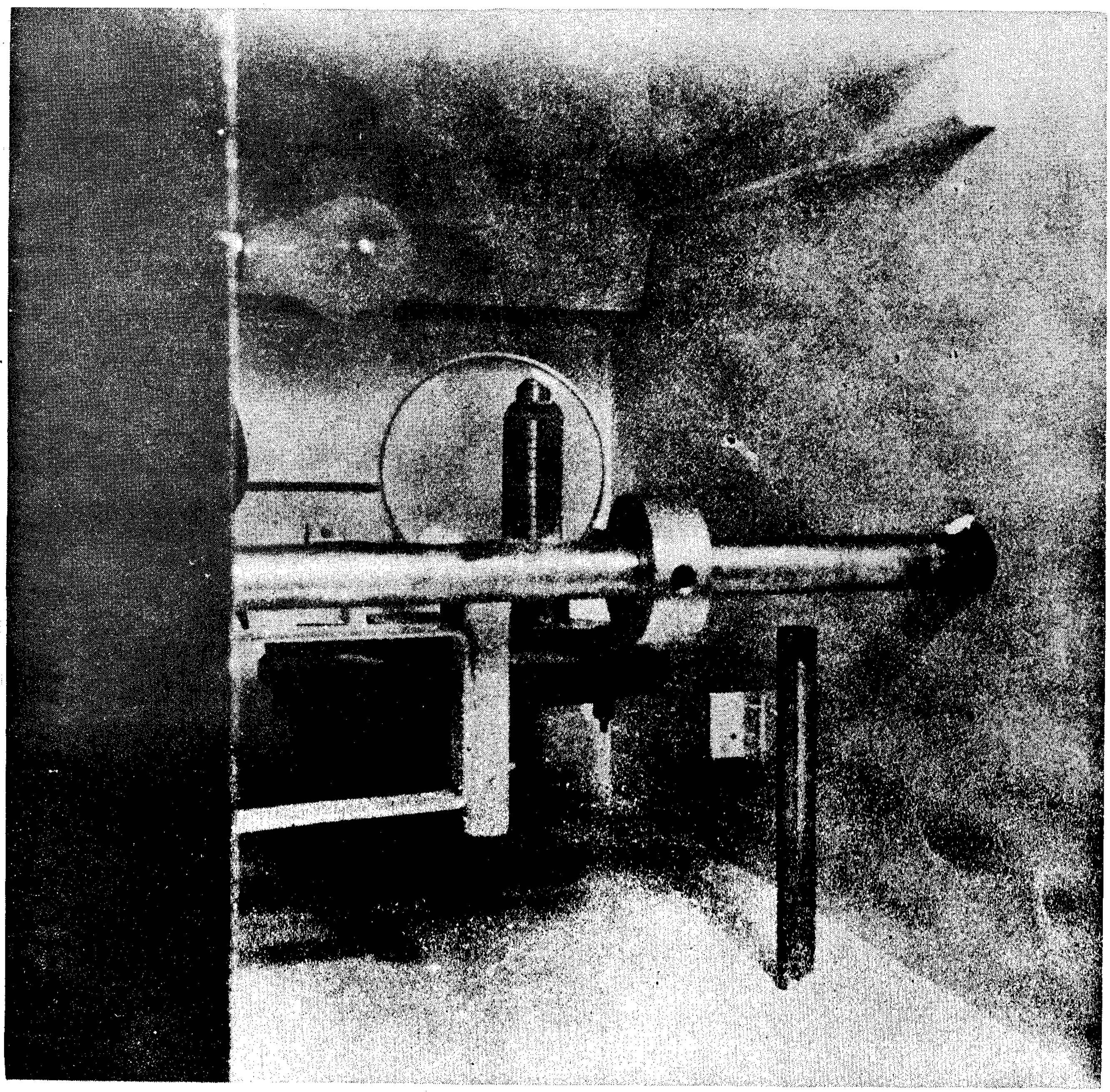
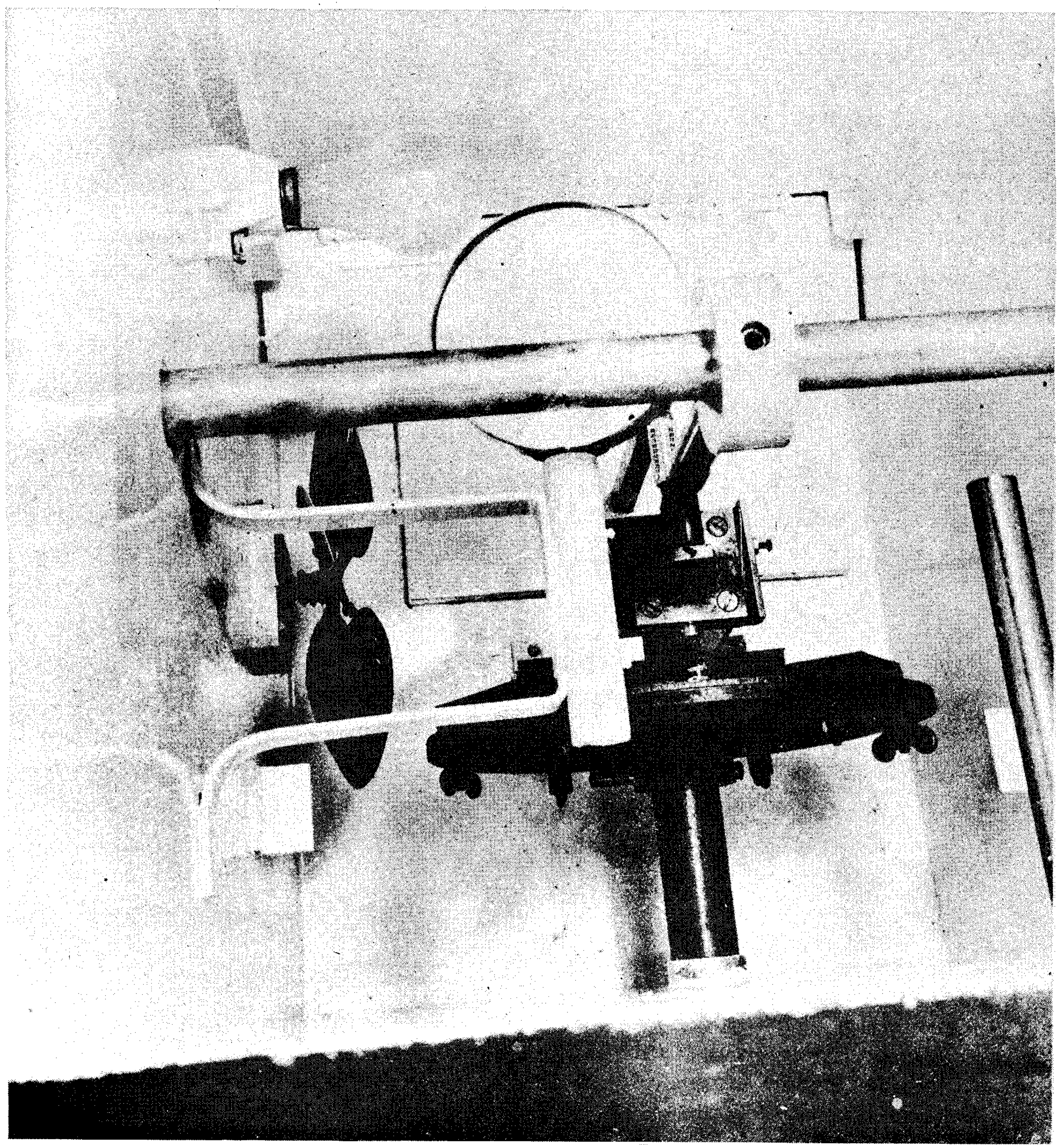


FIGURE 5513a



OBJECTIVES OF THE LOW TEMPERATURE TEST METHODS EVALUATION BY THE BUREAU OF SHIPS

By

T. A. Werkenthin

As you know, the Bureau has been active in low temperature testing for a number of years. Because there presently are numerous tests of the same property in rubber products, we have been attempting to determine which was the best method of determining each property. If it proves impossible to select one method which is superior to the others, we hope to be able to correlate the results which are obtainable by the two or three best methods.

In order to achieve this objective, the Material Laboratory of the New York Naval Shipyard has been engaged for over five years in evaluating individual test methods. To measure the applicability of these techniques to testing of the various synthetic rubbers, fifteen samples including compounds of neoprene, GR-S, nitrile rubber, butyl, thiokol, and Hevea were used in each evaluation.

Because of the amount of work involved in this undertaking, the Bureau suggested in 1950 that Committee E-I of the ASTM might be interested in cooperating in the project we had under way. However, in the time which has elapsed since this suggestion was made, we have completed a large proportion of the studies of the individual test methods. We are now approaching the time to attempt to interpret the results obtained.

With this very substantial background of information at hand, we feel that the most benefit might be obtained by an intensive study of the data already available, rather than to undertake an entire new program. For example, we have

reports of each of the five common methods of determining hardness, all conducted under the same conditions, upon the same wide spread of materials. Our statistical experts are now studying the data, to determine their significance. We hope that at least some answers to correlation of hardness tests will be supplied by these studies.

At the present time, there are 30 reports available which describe completed evaluations of test methods. These cover the following:

HARDNESS TESTS

4855-Prel. #4	8-21-46	Shore "A" Durometer
" 9	2-3-47	" " "
" 5	9-6-46	Rex Hardness Gauge
" 8	1-10-47	" " "
4855-2 Final	8-20-49	British Admiralty Hardness Meter
-5 "	10-29-47	P & J Plastometer
-6 "	4-29-48	ASTM Hardness Tester

MISCELLANEOUS

4855-Prel #7	10-18-46	Constant Deflection Compression Set Apparatus
4855-8	10-12-48	" " "
-5	6-28-49	Comparison of Precision of 10-sec vs 30-min Determinations of Compression Set
4855-Prel #11	5-1-47	Cyanamid Brittle Point
" " 12	7-1-47	Bashore Resiliometer
4855-3	11-1-48	MacDonald Hi-Po-Log Strain Gauge
" -9	11-7-50	Yerzley Oscillograph

MISCELLANEOUS (cont'd)

4855-11 Prog. 1	7-13-51	Plying of Specimens, Effect of
" -13 Final	9-29-48	Linhorst Autographic Stress-Strain Harmonic Tensiometer
" - 27	6-28-49	Sealing Pressure Tests
" - 29	10-14-49	Bakelite Brittleness Tester
" - 30	3-9-50	U.S. Rubber Co. Retraction Test

FLEXING AND STIFFNESS

4855 Prel. #1	4-22-46	Olsen Stiffness, 40-inch-pound
4855-7	10-17-50	" " " 6
4855 Prel. #2	5-2-46	Firestone Flexure Apparatus
" " #6	10-2-46	Cantilever Beam Flexural Test
" " 10	3-4-47	Chemical Warfare Service Flexibility Method E-1
4855-4 Prog. 1	8-16-49	Gehman Torsional Test
" Final	1-19-50	Effect of Pre-treatments upon stiffness
4855-10	10-31-50	Gurley Stiffness Tester
" -14	7-14-48	Torsional Set Apparatus
" -15	7-13-51	Bakelite Torsion Tester
" -16	9-26-51	Aminco Modulimeter

Probably most of you already have received these reports, but extra copies can be furnished if desired. In addition to these completed reports, the Material Laboratory of the New York Naval Shipyard has under way nine additional projects studying various test methods.

Accordingly, the Bureau considers that the time is nearly at hand when the best test methods can be selected, on the basis of information now at hand. We are not in accord with the desires of ASTM to undertake a whole new program of evaluation.

In selecting a few methods for specification use from the large number now active, it is obvious that many methods which agencies presently rely upon must be discarded. Probably each of us will have to sacrifice some of our "pet" methods.

Mr. Lichtman from the Material Laboratory will give you a review of the Bureau of Ships program, and details of what has been accomplished to date. At present, we feel that the following methods should be considered for possible specification use:

1. Hardness - Pusey and Jones or modified Admiralty indentometer
2. Compression set - Constant deflection method
3. Flexibility - Cantilever beam, Clash-Berg, and Gehman methods
4. Resilience - Bashore Resiliometer

In conclusion, we should point out that while some test methods are entirely satisfactory for testing materials or differentiating between compounds, entirely different techniques may be needed to evaluate end items. While it is desirable to standardize as much as possible on specification tests, the great variation in specific and item requirements will make necessary a considerable number of tests for these specific tests.

METHODS OF TESTING ELASTOMERS AT LOW TEMPERATURES

Presented by

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INTRODUCTION

The Rubber Development Section of the Material Laboratory, as authorized by the Bureau of Ships, has been engaged for several years in the investigation of methods of evaluating the low temperature properties of elastomers. The purpose of this investigation was to permit the selection and standardization of the most suitable methods and apparatuses for incorporation in military procurement specifications. We are all cognizant of the importance of developing elastomers that are suitable for service at low temperatures and the parallel need for standardizing methods for evaluating elastomers under low temperature exposure conditions. The Laboratory has therefore concentrated a considerable effort towards the completion of its assigned task in this program.

A survey of the many low temperature evaluation methods and apparatuses in use generally or in individual laboratories permits most of them to be grouped into several broad classes, the grouping depending on the physical property being evaluated. The procedure to be employed in establishing the suitability of a material will then be determined by the manner in which it is used in service.

The first group of apparatuses include those used in determining the resistance of a specimen to deformation caused by application either of a constant or a variable

load. The second group consists of instruments used in determining the rate or amount of dimensional recovery of specimens after removing the deforming stress. The third group is composed of apparatuses employed in determining the brittleness or physical failure of a material subjected to rapid deformation while the fourth group includes devices used in evaluating changes in stress of an elastomer held under constant strain. Instruments not included in this paper are those used in evaluating the dimensional recovery of a specimen after removing a constant load and also instruments employed in evaluating changes in deformation (creep) of specimens held under constant load. It is believed that the latter properties are related to those classed in groups 2 and 4.

ELASTOMERS USED

The compounds used in most of the investigations consisted of a series of twelve stocks, two each of Hevea, Buna-S, Perbunan-26 Butyl, Neoprene and Thiokol-FA. As shown by the recipes given in Table I, one stock of each polymer type was compounded for optimum properties at normal temperatures while the second stock was especially formulated for low temperature service. Since these stocks covered a wide range in physical properties, they allowed an extensive comparison of the different instruments. In another cooperative program of work with the Office of Rubber Reserve, the Material Laboratory used a series of twelve stocks prepared by the Government Laboratory at Akron. These stocks, which are listed and described in Table II, were employed in investigating the correlation between the modified Gehman and Admiralty instruments.

EVALUATION OF RESISTANCE TO DEFORMATION

In surveying the methods of evaluation included in the first group, we find that a very considerable number of methods have been developed and reported. These methods may be further sub-divided on the basis of the manner of deformation of the specimen. Indentation of the specimen is employed by various hardness testers or indentometers as described in military, federal and industrial specifications (9), (10), (11), (12), (13), (23). The Shore Type A durometer, (14), (15), (16), the Rex Gauge (17), (18), the Pusey and Jones Plastometer (9) (12) (19) (23), The Admiralty Hardness tester (10) (20), an Admiralty hardness tester modified by the Material Laboratory to comply with the basic requirements of ASTM Method D531-49(9), and the ASTM hardness meter (22) were all investigated in our low temperature tests at temperatures down to -40F. Conditioning periods up to 94 hours at selected temperatures were used. The modified Admiralty indentometer (21) illustrated in Figure 1 was found to be the most satisfactory of the various indentation instruments investigated on the basis of its accuracy, simplicity of operation, rigidity and suitability for use at low temperatures.

Torsional deformation of a specimen by means of a torque load applied at the specimen ends is employed by the Gehman torsional tester (24) (25) (28), the Bakelite (Clash-Berg) torsional tester (26) (29) and the U.S. Rubber Company torsional tester (27). Investigations of these instruments indicated that the Gehman apparatus offered advantages over the other types with respect to

freedom from frictional errors and resulting improvements in the accuracy of the evaluations. The original Gehman instrument purchased from a commercial concern is illustrated in Figure 2. In order to improve the accuracy of deflection measurements and to insure non-freezing of the upper specimen grip, this instrument was modified as shown in Figure 3, some of the original accessory equipment was discarded where feasible. The multiple specimen mount was sacrificed for accuracy of allignment of the specimen grips, the specimen rack being replaced by a permanently fixed lower grip assembly and the upper grip being suspended from the torque wire as in the original instrument. By this arrangement, the zero pointer setting and specimen span length can be easily set and will remain adjusted during the course of a test. A check on the zero setting of the instrument is made by mounting a metal bar of rectangular cross-section in the specimen grips, making sure that the rotating head and specimen angle indicator are set to zero and noting the indicator reading after removing the metal bar from the grips. If the angle indicator remains at zero under these conditions, no twist was present in the wire and the instrument is properly adjusted in this respect. The distance between the specimen grips, which determines the span length, can be accurately adjusted by means of gage blocks.

A Dewar flask containing methanol and thermally regulated by immersion of dry ice chips therein was used in cooling specimens exposed to short time conditioning tests as shown in Figure 4. A calibrated Weton thermometer was used in lieu of the original thermocouple indicator and a stop watch was substituted for the electrical timer-light signal. A spirit level was attached to the base of the instrument to insure vertical allignment of the instrument and

a mirror was mounted over the torsion head assembly in order to facilitate reading the deflection scale when long time conditioning tests were made in a thermostatically controlled chamber. This chamber, which is shown in Figure 5, uses dry ice as refrigerant and was designed and constructed in the Material Laboratory for use in carrying out long time conditioning tests. As shown in Figure 6, the specimen conditioning chamber is in the upper half of the cabinet while the dry ice is charged onto the trays in the lower section. The blower in the lower section is thermostatically controlled while the upper circulating blower operates continuously.

The modulus of elasticity of rectangular cross-sectional specimens after short time exposure at temperatures down to -100F and after conditioning up to 94 hours at various temperatures was calculated by means of the Trayer and March (30) analysis on the basis of the degree of twist of a rectangular cross-sectional specimen of known dimensions and span length. In determining the change in modulus of a stock, in which the same specimen is evaluated under different conditions, simplified modulus proportionality factors based only on the degree of twist may be determined to indicate the degree of change of modulus. The ratio of the modulus proportionality factor of a specimen determined at a particular base temperature, such as 75F, to that determined after exposing the specimen at some low service temperature may be used as an index of the relative suitability of the material under low temperature service conditions. The selection of the temperature and period of exposure will depend largely on the expected service requirements for the material.

The relationship between constant load hardness indentation and the flex modulus of rubber materials has been investigated by J. R. Scott (31) (32). Scott found that this relationship may be expressed by the equation, $E=A/H^{1.35}$.

In this equation "A" is a constant determined by the physical characteristics of the instrument, namely, the indentor and the major load. It follows from this equation that, for a particular instrument using the same indentor and major load in all tests, a modulus proportionality factor equal to E/A may be expressed by the equation, $E/A=1/H^{1.35}$.

The relationship between flex modulus or stiffness and hardness indentation expressed by this equation was investigated using the Material Laboratory modification of the Admiralty Indentometer and the modified Gehman tester. Specimens prepared from the Office of Rubber Reserve stocks listed in Table II were conditioned for 94 hours at temperatures ranging from 75F to -50F. The values of flex moduli of the various stocks were calculated from the Trayer-March equation and the Gehman instrument data and are presented graphically in Figure 7. The sodium-catalyzed 75/25 butadiene-styrene material, an unplasticized Perbunan-26 compound (No. 394-S-7) and the 14F polybutadiene compound all showed considered larger increases in modulus at progressively lower temperatures than the other materials which included a plasticized Perbunan-26 stock (No. 394-S-12), a 122F polybutadiene stock and plasticized and unplasticized GR-S stocks. The plasticized GR-S and the plasticized 122F 85/15 Bd/S stocks showed the lowest increases in modulus. Values of flex modulus and hardness indentation of the compounds determined at comparable

intervals after deformation were plotted as shown in Figure 8. The semi-log plot shows the relationship between flex modulus and hardness indentation to be represented by a hyperbolic function of the form $Y=Cx^n$. A log-log plot of the data over the range of indentations from 1.7 to 0.1 mm and of flex modulus from 350 to 20,000 psi is shown in Figure 9. Evaluation of the plot shows the relationship to be expressed by the equation $E=820/H^{1.36}$. This equation is similar in form to that determined by Scott, the exponent being very close, and it shows the basic equivalence of flex stiffness and resistance to indentation. Both characteristics are manifestations of the same basic property, namely resistance to deformation.

Other methods of evaluation of flex stiffness were also investigated in the Material Laboratory or at Laboratories in which the instruments were located. Those instruments in which the specimen was deformed as a centrally loaded end-supported beam or as an end-loaded cantilever beam included: (a) The Liska apparatus (33)(34)(35)(36) a modification of which, manufactured by the American Instrument Company, was also investigated (37), (b) the Tinius Olsen stiffness testers of 40 in. lb. and 6 in. lb. capacities (38)(26) (40)(41)(42), (c) the Werkenthin cantilever flex test presently specified as a method of evaluation of the low temperature properties of gasket materials (23)(43) (44)(45), and (d) the Gurley Stiffness tester manufactured by W. and L. Gurley, Troy, New York (48). The Tabor stiffness gauge, which is similar to the Tinius Olsen stiffness testers, in which cantilever specimen is deflected to a

known degree against a pendulum of adjustable load is presently being investigated in the Material Laboratory.

The MIT flex tester (46)(47) was also reviewed for investigation but was not considered suitable for general specification use in the evaluation of low temperature properties of elastomers and elastomeric products. The instrument was designed for use in determining the radial compression of a ring type specimen of prescribed dimensions under constant load. As such, it may be considered to be a specialized modification of the Liska type of apparatus and was therefore not investigated further. Although some of the instruments had individual advantages, such as the variability of adjustment of specimen span or deflecting load, the modified Gehman torsional tester was found to offer the most advantages in respect to freedom from friction of the load measuring components, accuracy of time of reading after deformation of the specimen, ease of manipulation of the instrument, compactness of the instrument and small size of the specimen. The latter characteristic is important since a specimen of small cross-section would be more suitable for solvent immersion conditioning prior to low temperature testing than a specimen of large cross-section.

Also included in the investigation (49) was a recording stress-strain tensiometer developed by Morron, Knapp, Linhorst and Viohl (68). This device permits the determination of tensile modulus of a specimen at 25% elongation and evaluation of hysteresis characteristics under repeated extension and retraction. The instrument operation and analysis of the data are affected, however, by inertia of the moving parts.

In view of this, the device was not considered as suitable for specification use as the modified Admiralty or the modified Gehman instruments.

The low temperature tensile modulus method of evaluation described by Graves and Davis (59) has not, as yet, been evaluated by the Material Laboratory. Their method employs a conventional Scott tensile machine having a Dewar flask and a goose-neck type clamp mounted on the lower cross-head. This arrangement permits a specimen to be elongated while it is immersed in a liquid medium at the desired low temperature. The machine is operated at a jaw separation speed of 12 ipm in making a measurement of modulus at a specimen elongation of 25%. The increase in modulus of the material at -25C in comparison with the modulus at +25C is used as an index of the low temperature serviceability of the material. The method appears to be precise and may therefore have merit for use in short time exposure tests on elastomers. However, refinements in the apparatus should be made and extreme precautions in the procedure must be taken in order to insure accuracy of long time conditioning exposure tests. The elongation evaluation method described by Morris, James and Evans (67) employs T-50 specimens which are evaluated under a pre-determined stress in order to determine the change in elongation of the material with temperature of exposure. The apparatus, although reasonably simple and accurate in operation, does not possess the additional advantages of rapid adjustment of exposure conditions and compactness and simplicity of the load measuring components indicated by the Gehman torsional apparatus.

EVALUATION OF DIMENSIONAL RECOVERABILITY

The second group of methods investigated by the Material Laboratory are intended for use in determining the rate and amount of dimensional recoverability of specimens after conditioning them under constant deformation. Although any manner of deformation may be employed such as compression, elongation, torsion, indentation or bending, the first is used in the majority of methods or apparatuses in this group.

The methods of evaluating compression set are generally based on ASTM method D395-49T (50), method B providing for conditioning of a specimen compressed to a definite degree between parallel plates and determination of the degree of recovery of the specimen at a definite time after removal from the compression plates. Differences in these methods are minor and consist of variations in the conditioning medium used, that is, air, carbon dioxide and air, methanol or other liquid, or differences in design of the plates or the compressing mechanism. It was attempted in the first phases of the investigation to compress the specimens after conditioning them at the selected low temperature. This procedure was abandoned however due to the difficulty of assembling the plates at low temperatures under the high loads required to compress the frozen specimens. The procedure finally developed (51) provided for the compression of the specimen at room temperature after which the jigs are exposed to the selected temperature conditioning. The jigs are disassembled and the recovered dimensions of the specimens are measured in the conditioning chamber at specified time intervals. This procedure has been included in a military gasket

specification (43) and is now being considered by ASTM Sub-Committee XVII as a tentative standard for low temperature evaluations. The procedure is considered a highly significant one for evaluation of dimensional recoverability, as the test simulates a condition often encountered in service such as in a door, hatch or scuttle gasket.

Investigations of a tensile retraction apparatus similar to those reported by Svetlik and Sperberg(52) (55) and Labbe and Schade (53) were also conducted. In addition, photographs of a tensile retraction apparatus designed by personnel of the General Laboratory, U.S. Rubber Company (56) were used in the construction of the Material Laboratory model. The Material Laboratory apparatus permitted the evaluation of seven T-50 type specimens simultaneously at elongations up to 250%. The specimens are racked at room temperature and then they are immersed in a methanol bath at -94F and allowed to condition for 10 minutes. Following this, the specimens are released while in the bath and allowed to recover as the bath temperature is raised at a rate of 1C per minute. The temperature of the immersion bath is measured at particular specimen retractions during this time. Specimens were also conditioned at -40F for 10 minutes while elongated and then released; the retraction being measured 1 minute after release. The bath temperature is held constant during the retraction period.

The data obtained in the investigations by Svetlik and Labbe were used to determine a freeze point or temperature corresponding to a 0% retraction. This point was compared to the Gehman freeze point obtainable by a plot of the mod-

ulus proportionality factor or specimen twist angle vs. conditioning temperature. In the Material Laboratory investigation (54), however, the tensile elongation apparatus was investigated to determine the feasability of its use in evaluating the set properties of an elastomer at low temperatures.

A correlation was found between the 10 sec. compression set determinations for specimens conditioned 1 hour at -40F and both the 70% retraction temperature and the percent retraction of specimens measured 1 minute after release and test at -40F. The tensile retraction type of apparatus does not appear to offer any distinct advantage over the compression set apparatus in the evaluation of the deformational recoverability of a material. It is therefore proposed that the latter procedure be continued in use for the determination of this property of elastomers.

EVALUATION OF BRITTLENESS

In surveying the methods of evaluation of the third broad property of elastomers, namely brittleness or visible failure of a material when subject to rapid deformation, we find that although there are a number of different apparatuses designed to evaluate this property, they are all generally in conformity with the procedural requirements of ASTM tentative method D 746-44T (57). Despite the structural differences between the several brittleness testers described by Graves and Davis (58)(59), Salker, Winspear and Kemp (60), Smith and Dienes (61) and Bimmerman and Keen (62), they all cause a rapid deformation of a cantilever specimen by a striker moving relative to the specimens. Compliance of these aparatuses whether mechanically, spring, or solenoid-operated,

with the basic requirements of ASTM method D746-44T is governed by the relative velocity of movement of the striker, past the specimen, the shape of the striker edge, the free span of the specimen before deflection and the specimen dimensions. The Material Laboratory investigations of the Graves-Cyanamid apparatus (58)(63) and the Smith-Bakelite apparatus (61)(64) showed the brittleness temperatures obtained by means of the former device to be somewhat lower than those obtained using the latter possibly due to the smaller cross-sectional dimensions of the specimens used in the Graves apparatus. It is believed that any apparatus in conformity with the basic requirements of the ASTM method D746-44T and used in accordance with the procedures described in this specification would be suitable for evaluation of the brittleness of elastomeric materials. Variations in the kinetic energy of the moving components of the instrument at the time of impact may account for variations in the results obtained using different apparatuses otherwise conforming to the basic requirements of the ASTM specification.

EVALUATION OF STRESS RELAXATION

Evaluation of the rheological properties of elastomers or their stress-strain relationships over a period of time may be conducted by evaluation of the degree of creep or the stress relaxation of the material. The creep of a material is indicated by its deformation under constant stress while the stress relaxation is indicated by the change in stress of the material under constant deformation.

Although creep may be evaluated by use of simpler instrumentation than that required in the evaluation of stress

relaxation, as many of the service applications involve the stressing of the elastomer under constant deformation, the methods discussed in the present paper will be those concerned with the evaluation of stress relaxation.

The apparatuses and procedures used in evaluating stress relaxation are generally based on two methods of deformation of the specimen, those employing a compression or indentation deformation of the specimen and those using a tensile deformation. In the first group are apparatuses using strain gage detecting units as reported by MacDonald and Ushakoff (6), and Phillips and Labbe (3), variable load beam apparatuses reported by Blow and Fletcher (2) and Beatty and Juve (8), compressed-air loading apparatuses reported by Wilkinson and Gehman (1) and Morris, James and Seegman (4) and the sealing pressure apparatus described in various military specifications (43)(44) and ASTM tentative method D1081-49T (66). The apparatus reported by Tobolsky, Prettyman and Dillon (7) on the other hand is based on tensile deformation of the specimen.

Where possible, investigations were conducted by the Laboratory on the original models of the apparatuses. It was necessary in most instances, however, to construct models of the original apparatus in the Material Laboratory. In such cases, changes were made in the design where feasible to facilitate the simultaneous evaluation of a larger number of specimens with a limited expenditure and to facilitate the exposure of the specimen to various conditioning media and environments during or prior to deformation.

In most of the Material Laboratory investigations the stress relaxation characteristics of the compressed specimens were determined over a period of 22 hours. Wherever possible, the zero time, or time of determination of the first stress reading after deformation, was .01 hr. Although some investigators have made stress relaxation determinations over considerably longer periods of time, up to 300 hrs. or more, the shorter period of 22 hours was considered suitable for determination of the correlation among the instruments and their suitability for general laboratory use in evaluating stress relaxation of elastomers at low temperatures. Since the major degree of stress relaxation of an elastomer may be expected to occur within a relatively short time after deformation and as a shorter time of deformation will permit many more determinations to be made, the shorter conditioning cycle is preferable in attaining rapid completion of the program and adoption of suitable procedures.

The original apparatus constructed by MacDonald and Ushakoff was investigated in the Material Laboratory. The investigation (69) although limited by the time of loan of the instrument showed evidence of a relatively sharp break in the stress decay curve for a natural rubber stock exposed for about 94 hours at -35F, probably due to a change in the specific volume of the material associated with crystallization. Further investigations are now being conducted in the Laboratory employing the strain gage apparatus illustrated in Figure 10. A twelve channel switching unit is being used to permit the conditioning of twelve jigs simultaneously. The

individual jigs are very similar in design to those described by MacDonald and Ushakoff, a diagramatic sketch of which is shown in Figure 11. Results obtained in the preliminary phases of the present investigations have been plotted as S/So ratios versus log time where t_o=0.01 hours after start of compression. Graphs of representative curves for 12 stocks are shown in Figures 12 and 13 for normal type compounds and cold resistant compounds, respectively. Further investigations are now underway using this apparatus to evaluate stress relaxation properties of elastomers at low temperatures. In this connection, it may be necessary to modify the jigs or to reduce the size of the specimens if it is desired to test specimens that have been subjected to low temperature conditioning, before they are compressed. Such an investigation would be of considerable interest and should be undertaken after a suitable instrument and precise procedures have been developed.

The Phillips and Labbe strain gage stress relaxation apparatus shown in Figure 14 employs Baldwin SR-4 Type C load cells as the load detecting element. The lower movable platform is elevated by means of a crank and gear mechanism in order to compress the specimen between the upper load cell and the lower platform. The degree of compression is indicated on the graduated dial and counter. The specimen stress which causes deformation of the load cell is transmitted to the strain gage circuit and hence to the Foxboro type recorder. This apparatus is essentially comparable to the previous ones except for the specimen deflecting mechanism. Owing to the large size and weight

of the jigs and the resulting difficulty in handling them, the apparatus was not investigated further by the Material Laboratory. Instead, work was concentrated on the MacDonald type of loading and deflecting jig.

Although the strain gage type of stress relaxation apparatuses have particular advantages over the others in regard to the rapidity of recording of the initial stress characteristics, this may not be of considerable significance in evaluating the service performance of a material when the stress retention after a relatively long deformation time may be much more important than the stress characteristics after a short strain period. In deforming an elastomeric material in the usual type of service, the period to strain the material may be as much as a minute or more during which time the material will be undergoing relaxation. A zero time of from .01 to .1 hours would therefore be appropriate. The accurate determination and standardization of the zero time is important however in a laboratory evaluation regardless of its magnitude.

The variable load beam apparatuses designed by Blow and Fletcher (2) and Beatty and Juve (8) are similar to the strain gage apparatuses in respect to the manner of deformation of the specimens, the round plug type specimen being compressed to a known deformation and held at this deformation during the test period. The manner of evaluating the specimen stress reaction to deformation is a mechanical one however employing a variable weight on a beam to balance the specimen stress. A signal system is used to indicate the balance point without a significant change in deformation of

the specimen. The Material Laboratory has completed construction of an apparatus essentially equivalent to the Blow-Fletcher and Beatty-Juve models. This apparatus is illustrated in Figure 15. The round plug type specimen is clamped between the compression plates using a vice or air press to achieve rapid compression. Separator blocks are used in order to deform the specimen only a predetermined amount, 40% being used in the preliminary tests.

The jig is similar to that proposed by F. C. Thorn of the Garlock Packing Company, Palmyra, New York and is essentially similar to the constant deflection compression set jig described by ASTM Tentative Method D 395-49T (50). The assembled jig is transferred and mounted in the micrometer section of the load indicating apparatus. The micrometer platen is then lowered until the signal indicates the beam to have been depressed to contact the microswitch. At this point, a very slight further downward movement of the beam would cause an alternate signal, the degree of downward travel being controlled by an adjusting bolt located under the beam near the microswitch. The compression bolts of the jig are then backed off and the spacer bars are removed. As soon as the bolts are backed off, the specimen stress causes the beam to move downward minutely and activate the alternate signal. The dynamometer is then elevated by means of the hand wheel or a turnbuckle or other elevating mechanism until the first signal is obtained at which point the specimen stress is in balance with the dynamometer load. Since the instrument physical dimensions are constant, the dynamometer readings may be used direct-

ly in the determination of the stress ration characteristics S*/So. The spacer bars are then replaced in the jig and the compression bolts are tightened. Following this operation, the jig may be removed and subjected to further conditioning in liquid or other media and at various temperatures, the specimen stress being determined at desired intervals thereafter. The Laboratory is completing adjustment of the instrument and will begin evaluation runs at normal and at low temperatures in the near future. Although a non-recording apparatus such as this will make the t_o interval somewhat longer than the strain gage type of apparatuses this interval has been estimated to be only about 1 to 2 minutes between the time of compression of the specimen and balancing of the dynamometer. The significance of a change in zero time which of course will be constant for all tests made with the particular apparatus, in comparison to a 36 sec. zero time will depend on the accuracy and precision of the data obtained in the respective investigations. The advantages of the beam type apparatus, such as lower cost, simplicity of the mechanism and ease of conditioning of the compressed specimens will also be discussed at the conclusion of these investigations.

A compressed-air-loaded apparatus being used in the investigation of stress relaxation evaluation apparatuses at low temperatures was based on the apparatus described by Wilkinson and Gehman (1), and shown in Figure 16. The instrument is designed to indicate the progressive decrease in load required to maintain a specimen under constant deformation. Unlike other apparatuses, such as that reported by Blow and

Fletcher, Beatty and Juve, and MacDonald and Ushakoff, the Gehman type apparatus incorporates an air bellows to maintain an automatic, self-regulated balance between the compression load indicated by bellows pressure and the specimen compressive stress.

The apparatus constructed in the Material Laboratory is shown in Figures 17 and 18. Air pressure is used to furnish the force to compress the specimens. A Schrader type inlet valve is mounted in a surge tank at the upper part of the apparatus. An expansion bellows supported under the upper plate of the apparatus and connected to a pressure gage and to the surge tank functions as a compression piston acting on the specimens placed on the upper surface of the central plate. A Schrader valve mounted in the bottom plate of the bellows acts as the pressure-stress control valve. An adjustable cross-bar is positioned so that on admission of air pressure to the bellows and subsequent downward movement of the bellows the specimens are compressed to a predetermined amount. The amount of compression or deflection of the specimens is indicated on a dial indicator supported below the center plate and contacting the lower surface of the aluminum loading block between the bellows and specimens. The position of the cross-bar is adjusted so that, at the desired deflection, the trigger pin of the Schrader valve in the bottom of the bellows is just in contact with the cross-bar and any further downward movement of the bellows and loading block against the specimens would results in opening of the valve and leakage of air from the bellows. On relaxation of stress in the specimens under the loading

block, the bellows tend to expand and thus move downward. The movement however results in opening of the control valve as indicated and leakage of air from the bellows until the specimen stress is again in balance with the bellows pressure, at which point the valve will again be closed. The cyclic adjustment of specimen stress and bellows pressure continues through the test period the bellows pressure at any time therefore being indicative of the specimen stress.

Six specimens were used in each run spaced equally under the loading block to insure equal loading and deflection of the specimens. The number of specimens used in each run would be dependent on the compression modulus of the material. At low temperatures it may be necessary to decrease the number of specimens in order to maintain the same deflection and bellows pressure range. The cross bar was adjusted to produce a compression of 40% for specimens of 1/2 inch nominal thickness. The pressure readings were taken from .01 hours after compression of the specimens to 22 hours, after compression, making duplicate runs. The results of evaluations of the standard stocks at room temperature are shown in Figure 19. Results of evaluations of the low temperature service stocks are shown in Figure 20. It is seen from these charts of S_t/So versus log time that the hevea stocks show least relaxation while the Perbunan-26 stocks show relaxations of as much as 30-34% in the same period. The increases in the rate of stress relaxation may be influenced at room temperatures by oxidation and chanin scission. At low temperatures these factors may be expected to exert little or no influence, being replaced in importance by crystal-

lization, specific volume changes and changes in internal viscosity of the stocks.

The accuracy of the Autopneumatic apparatus is dependon the efficiency of operation of the lower control valve. Improper leakage of this valve will, of course, result in inaccurate determinations. Since low temperature exposure in particular may result in inefficient operation, the feasibility of running the low temperature tests using a very small but constant air flow slightly larger than the leakage anticipated from the valve in the closed position will be investigated. In this manner the control valve will be caused to float at some definite displaced position, the possibility of a leakage error thus being compensated. The rate of air leakage may be controlled by means of a flow regulator and indicator.

The autopneumatic apparatus shows other disadvantages. For example, only one stock may be evaluated during a test period and specimens cannot be readily exposed to various conditioning atmospheres or media while mounted in the instrument.

The compressometer designed and reported by Morris, James and Seegman (4) also employs air as the loading medium, the specimen likewise being deformed in compression. The apparatus has not yet been included in the Material Laboratory investigations due to further modifications by the original designers. The instrument consists essentially of an air-operated piston for compressing the rubber specimen and a dial indicator for measuring the degree of compression. The piston travel may be adjusted to obtain a definite degree

of compression. To determine the specimen stress at any time, the air cylinder is loaded until the indicator shows a very slight downward movement of the piston. The specimen stress may be determined if desired on the basis of the indicated air pressure and the physical dimensions of the specimen and piston.

The NRL - Precision sealing pressure apparatus illustrated in Fig. 21 is presently being used in the Material Laboratory to evaluate the pressure sealing properties of gaskets procured under specifications MIL-R-900A (43) and 33R9 (44) and is described in these specifications and ASTM tentative method D1081-49T (66). The specimen is indented a specified amount, usually 1/16 in., by means of the spherically tipped indentor. This is accomplished by rotating the base upward relative to the indentor stem. The stem is drilled from one end to the other to form an air passage and is connected to a controlled source of air during a sealing pressure determination. In preliminary investigations of the suitability of this apparatus for use in evaluating stress relaxation properties of elastomers at low temperatures the specimen was found to be cut by the indentor when indented after low temperature conditioning and to cause plugging of the indentor. In subsequent tests the specimens were indented rapidly at room temperature and immediately placed in a thermally controlled bath at the desired temperature, this procedure resembling that used in the preliminary investigation (69) of the MacDonald apparatus. Short time relaxation runs of 1 hour duration were made at temperatures from 75 to -35F while long time stress relaxation tests were made

at -20 and -35F over a 94 hr period. The jigs were transferred from the alcohol bath to a thermally regulated dry ice conditioning chamber for the long time conditioning tests, methyl alcohol being used as the leak indicating fluid at low temperatures in the conditioning chamber.

Sealing pressures were determined 1 min., 30 min. and 60 min. after indentation and immersion of the jig in the regulated bath and 46 and 94 hrs. after transfer to the conditioning chamber.

The stress relaxation was considered to be indicated by the ratio $P_t/_{po}$ where P_t was the sealing pressure determined after 1 hr., 46 hr. and 94 hr. and P_o was the sealing pressure determined 30 minutes after indentation of the specimen. The sealing pressure characteristics over a 94 hr. period at -20F are shown in Fig. 22. A gradual drop followed by an increase in the pressure ratio was obtained in tests of a number of compounds including Perbunan-26 No. E-194-388 and #-194-224, Neoprene No. E-156-290 and Buna-S No. E-162-415 while several other compounds including Thiokol-FA No. E-53-15 and E-53-17 and Hevea Nos. E-13-92 and E-13-173 show a decrease in the pressure ratio. The decrease of the ratios of the latter compounds may be due to crystallization and associated decreases in specific volume, as well as stress relaxation. The increases in the ratio value with time of the former compounds may be due to adhesion of the indentor to the specimen or gradual plugging of the instrument. The data indicates an increase in relaxation of some crystallizable compounds in contrast to an absence of relaxation shown by the GR-S, Perbunan-26 and

neoprene compounds.

This conclusion is confirmed only partially by the long time evaluations at -35F as shown on Fig. 23. The Hevea and Neoprene compounds show increases in pressure ratio while the Thiokol FA and Butyl compounds show decreases. Stress relaxation evaluated on the basis of the pressure ratios at low temperatures was found to be influenced by various factors including the occurrence of crystallization of the polymer or other components and also possible plugging or adhesion of the specimen to the indentor. Thus Buna-S stocks which would not be expected to show any significant changes in internal viscosity or stiffness during a long time conditioning period nevertheless did not show a linear pressure ratio - log time characteristic.

The possibility of correlation of stress relaxation and compression set properties of elastomers has been investigated by Wilkinson and Gehman (1) and Beatty and Juve (8). These investigations have shown this correlation to be only fair. The compression set property may be considered to be essentially the measure of the ability of a deformed elastomer to recover its original dimensions after removal of the deforming stress, the recoverability being dependent on the retained molecular stresses attempting to produce recovery and factors resisting recovery such as the internal viscosity and the presence of crystallized orientations of the molecules. It was decided to evaluate this recoverability by determination of the sealing pressures before and after a slight separation of the specimen and indentor rather than by a measurement of the specimen stress

at widely separated time intervals as would be indicated by the stress relaxation data. The shorter interval would correlate with that used in evaluating the compression set. The separation was made at the rate of .005 inches per minute immediately after taking the 1 hour sealing pressure measurements in the 1 hour conditioning evaluations at temperatures down to -35F and after the 46 hr. and 94 hr. conditioning periods in the long time evaluations at -20F and -35F. Three specimens were used in each determination at each temperature and each conditioning period to avoid recompression of specimens after retraction. Pressure readings were taken after each .005 in. back-off, although it was found that the reading after three min. retraction corresponding to .015 in. total retraction was the most useful, differentiating between those stocks showing high and low recovery. Ratios of sealing pressures after .015 in. total back-off and immediately before the start of retraction, that is, zero retraction were calculated and plotted.

The degree of pressure retention with decrease in conditioning temperature is shown in Fig. 24, indicating a general decrease in pressure retention with decrease in temperature, the rate and degree of decrease being dependent on the compound. The Thiokol-FA compounds E-53-15 and E-53-17 show the most rapid decrease in stress retention; the Perbunan-26 compounds being slightly better, and the Hevea compounds showing the best retentivity. Data for the long time conditioning tests made at -20F. are shown in Fig. 25. These data show a decreasing retention ratio for almost all compounds evaluated, the Buna-S compound

E-162-489 and Butyl compound E-34-104 showing the least decrease. The other compounds showed decreases with conditioning time of varying degrees, the Thiokol-FA compounds and Perbunan-26 compound E-194-388 showing the poorest stress retention due to the extensive crystallization and to the high internal viscosity. The long time conditioning tests at -35F show essentially the same decrease in pressure retention with conditioning time as the -20F tests as shown in Fig. 26. Neoprene stock No. E-156-290 exhibited a lower retention at -35F than at -20F. The Buna-S compounds showed no significant change in degree of retention except for the 46 hr. value of stock No. E-162-415, which point is considered questionable. Perbunan-26 stock No. #-194-224 showed a lower retention at -35F than at -20F and the remaining stocks showed only small changes, if any, in the rate of change of stress retention with conditioning time in tests at the two temperatures.

The relationship between the scaling pressure ratios and compression set data determined under essentially the same conditions is shown in Fig. 27. No distinction is made in regard to the manner of conditioning the many specimens. Although a considerable scattering of the data is indicated, a correlation between the stress retention and compression set is apparent. A low pressure retentivity corresponds to a high compression set and vica versa. The stress retentivity as evaluated at .015 in. back-off may not be directly related to the stress relaxation characteristic of a material as the retentivity value is dependent on the internal viscosity, molecular crystallization or other deterring forces acting in the material. The stress relaxation would however be determined ideally only be evaluating the stress in a material without inducing any change of deformation on the material at the moment of measurement.

This requirement of dimensional stability in the evaluation of stress relaxation is met by the strain gage types of apparatuses and others in which the specimen is permitted to act against the stress measuring system without any recognizable change in deformation.

The tensile stress relaxation apparatus reported by Tobolsky, Prettyman and Dillon (7) is based on the use of a ring type specimen deformed in tension to a known elongation. Although the original investigations using this apparatus were directed toward the study of oxidative changes in elastomer chanin structures and their effects on stress relaxation, this apparatus may also be used in the evaluation of stress relaxation properties of elastomers at low temperatures at which such effects would be very small.

The apparatus designed in the Material Laboratory for this investigation is shown in Fig. 28. The test specimen used is a T-50 type specimen in lieu of a ring type specimen, the T-50 specimen being more readily prepared. The stress in a T-50 type specimen is also not effected by roller friction and the possibility of non-equal stress in each side of a ring type specimen. The specimen is mounted in a jig which may be removed from the stress measuring component in order to permit conditioning of the specimen in various programs such as immersion in liquid media or other procedures. The load measuring devices used are Hunter spring gages of 500 and 5000 gm. capacity. The instrument is being adjusted and modified at the present time in the Material Laboratory in order to insure measurement of the specimen stress rather than a tensile modulus

at a particular elongation. It is proposed to plot the S/S_o values versus the log time as in other investigations and evaluate the correlation of the data with that determined in other investigations as well as the suitability of the instrument for use in evaluating the stress relaxation of elastomers after exposure under various conditions.

CONCLUSIONS

The investigation and analysis of the various instruments discussed will permit a selection of representative ones for specification use. The specification of particular devices does not, of course, in itself insure the procurement of materials suitable for the intended service. Of equal importance is the specification of the particular procedure and conditions to be used in a test. In this connection, it is possible to use most instruments under widely different conditions depending on the purpose of the evaluation. For example the torsional deformational apparatus and the tensile retraction apparatus may both be used in the determination of a freeze temperature by plotting the change of deformation on recovery of a specimen with change in temperature. The evaluation of this freeze temperature is generally determined in a short time exposure of the specimen. In the investigations at the Material Laboratory however the procedures used in the several tests were based on the intended use of the elastomer materials. For example, gasket compounds purchased under Military Specification are expected to afford a seal for extended periods of time at temperatures down to -35F. It was therefore necessary to design the investigational procedures around this service requirement. In investigation of

the methods of evaluation of all four groups of properties discussed, namely resistance to deformation under application of a load, the dimensional recoverability of a material after constant deformation, brittleness under rapid deformation and lastly the change in stress of a material under constant deformation, the suitability of the method for evaluation of the property of a material under long time exposure at temperatures of -20 and -35F was of primary importance.

On the basis of the results of the individual investigations and the conditions defined by the service it was found that a hardness indentation apparatus similar to the Admiralty Meter modified to comply with the requirements of Federal Specification ZZ-R-601a (12) and Bureau of Ships General specification Appendix I (23) or a torsional stiffness apparatus similar to that described by Gehman, Woodward and Wilkinson (24)(25) and modified as illustrated in Fig. 3. would be suitable for evaluating the resistance of elastomers to deformation under stress. The use of an indentometer has been adopted in Military specification MIL-R-900A (43) for evaluation of the change in resistance to deformation at low temperatures and may serve as a model for specifications of other elastomeric products requiring low temperature flexibility. A torsional apparatus may be more suitable for use in detecting changes in low temperature properties of elastomers due to previous conditioning by immersion, high temperature oven aging or other conditions due to the smaller cross-section of the specimen used. The indentometer has the advantage however of using a more easily prepared specimen and a simpler evaluation procedure.

In evaluating the dimensional recoverability of an elastomer after constant deformation, the procedure developed in these investigations and now specified in military specification MIL-R-900A (43), is one in which the specimen recovery is evaluated 10 seconds and 30 minutes after release after long time compression, the specimen thickness being measured at the conditioning temperature.

Although relatively few Naval specifications for rubber materials require an evaluation of the brittleness of the materials because of the static nature of most service conditions, this property is adequately evaluated by means of the ASTM tentative method D746-44T (57). The length of time and temperature of conditioning of the specimen would, of course, have to be established in accordance with the anticipated service conditions.

Recommendations concerning the suitability of the respective procedures for use in evaluating the stress relaxation properties of elastomers at low temperatures cannot be made at this time. Considerations however in regard to the ability to condition specimens while under deformation over extended periods of time and in various media or atmospheres will be of significance in evaluating the respective procedures. It is expected that sufficient progress will be made in the near future on this group of investigations to permit recommendation of a suitable procedure for specification adoption.

Although new methods and apparatuses superior to those discussed for evaluation of low temperature properties of elastomers will no doubt be developed in the future it is

believed that the ones recommended herein are suitable for immediate specification adoption and will serve to insure the procurement of elastomeric materials suitable for the important function assigned to them. These apparatuses proposed have in addition the important advantage of being available throughout the industry today and may be adopted for low temperature use with the addition, where necessary, of suitable conditioning facilities.

TABLE I, FORMULATIONS OF MARE ISLAND COMPOUNDS

HEVEA STOCK	Std. E-13-92	C.R. E-13-173
Smoked sheets	100.0	100.0
Zinc Oxide	5.0	5.0
Stearic acid	1.0	1.0
Cotton Seed Oil	2.5	2.5
Heliozone	3.0	3.0
Age Rite Resin D	1.0	1.0
Captax	0.5	
Altax	0.5	0.5
Tuads	0.5	
D.P.G.		0.1
Sulfur	0.75	3.0
P-33 (FT)	1.0	1.0
Total	115.75	117.1

Cure for $\frac{1}{2}$-inch thickness: 25 minutes at 287°F.

GR-S Stock	Std. E-162-415	C.R. E-162-489
GR-s	100.0	100.0
Zinc Oxide	5.0	5.0
Philblack A (HMF)	50.0	50.0
Naftolen 510	20.0	
Heliozone	3.0	3.0
Tributoxyethyl Phosphate		5.0
Plasticizer SC		5.0
Dioctyl Phthalate		5.0
Diisobutyl Adipate		5.0
Thionex	2.0	2.0
D.P.G.	0.4	0.4
Sulfur	0.6	0.6
Total	181.0	181.0

Cure for $\frac{1}{2}$-inch thickness: 25 minutes at 310°F.

PERBUNAN STOCK	Std. E-194-388	C.R. E-194-224
Perbunan 26	100.0	100.0
Zinc Oxide	5.0	5.0
P-33 (FT)	30.0	30.0
Statex B (FF)	30.0	30.0
Bardol	10.0	
Cumar P-10	10.0	
Tributoxyethyl phosphate		10.0
Plasticizer SC		10.0
Stearic acid	1.0	1.0
Heliozone	3.0	3.0
Tuads	3.0	3.0
Captax	2.0	2.0
Vandex	0.1	0.1
Total	194.1	194.1

Cure for $\frac{1}{2}$-inch thickness: 30 minutes at 310°F.

GR-M STOCK	Std. E-156-315	C.R. E-156-290
GR-M	100.0	100.0
Zinc Oxide	1.0	1.0
XLC Magnesia	4.0	4.0
Thermax (MT)	20.0	20.0
Paraffin	2.0	2.0
Neophax A	10.0	10.0
Circo L.P. oil	15.0	15.0
Stearic acid	1.0	1.0
Neozone A	2.0	2.0
Sulfur		3.0
	155.0	158.0

Cure for $\frac{1}{2}$-inch thickness: 25 minutes at 310°F in press, plus 60 minutes at 310°F. in open steam.

GR-I STOCK	Std. E-34-104	C.R. E-34-105
GR-I	100.0	100.0
Zinc oxide	5.0	5.0
Stearic acid	3.0	3.0
Philblack A	30.0	40.0
Circo L.P. oil	10.0	10.0
Tuads	1.0	1.0
Dibenzyl ether		10.0
Captax	0.5	0.5
Polyac	1.0	1.0
Sulfur	2.0	2.0
Total	152.5	172.5

Cure for ½-inch thickness:
35 minutes at 310°F.

THIOKOL FA STOCK	Std. E-53-15	C.R. E-53-17
Thiokol FA	100.0	100.0
Zinc oxide	10.0	10.0
Pelletex (SRF)	40.0	65.0
Stearic acid	0.5	0.5
Plasticizer SC		5.0
Tributoxyethyl Phosphate		5.0
Altax	0.3	0.3
D.P.G.	0.1	0.1
Total	150.9	185.9

Cure for ½-inch thickness:
45 minutes at 300°F.

Note: The second stock in each group is designed for cold resistance.

TABLE II, FORMULATIONS OF O. R. R. COMPOUNDS

394-S	394-S-1	394-S-2	394-S-3	394-S-4	394-S-5
			122°F	14°F	Na
	Natural Rubber	GR-S X-539	Polybd XP-148	Polybd XP-169	Polybd 81PC4-6
Polymer	100.	100.	100.	100.	100.
Statex B	40.	40.	40.	40.	40.
Zinc oxide	5.	5.	5.	5.	5.
Sulfur	3.	2.	2.	2.	2.
Altax	0.75	1.75	3.	3.	1.25
Stearic acid	3.		1.5	1.5	3.
PBNA	1.5				
Final Weight	153.25	148.75	151.5	151.5	151.25

	394-S-6	394-S-7	394-S-8	394-S-9
	Na 75/25 Bd/S	Perbunan 26	85/15 Bd/S XP-138 122F	80/8/12 Bd/I/S 41F
Polymer	100.	100.	100.	100.
Statex B	40.	40.	40.	40.
Zinc oxide	5.	5.	5.	5.
Sulfur	2.	1.25	2.	2.
Altax	1.25	1.75	3.	3.
Stearic acid	3.		1.5	1.5
PBNA				
Final Weight	151.25	148.00	151.5	151.5

TABLE II, FORMULATIONS OF O.R.R. COMPOUNDS

394-S-10 GR-S		394-S-11 85/15 Bd/S (XP-138) MF		394-S-12 Perbunan 26	
Polymer	100.	Polymer	100.	Polymer	100.
Philblack A	40.	Statex B	75.	Statex B	75.
Zinc oxide	5.	Zinc oxide	5.	Zinc oxide	5.
Heliozone	1.	Plastolein 9050	10.	Adipol BCA	10.
Flexol TOF	20.	Dicapryl Sebacate	10.	Dibutyl Sebacate	10.
Methyl Tuads	0.8	Stearic acid	1.	Plasticizer 3425	10.
Sulfur	1.	Methyl Tuads	1.	Stearic acid	1.
		Sulfur	1.	Thionex	0.3
				Sulfur	1.5
Final Weight	167.8		203.0		212.8

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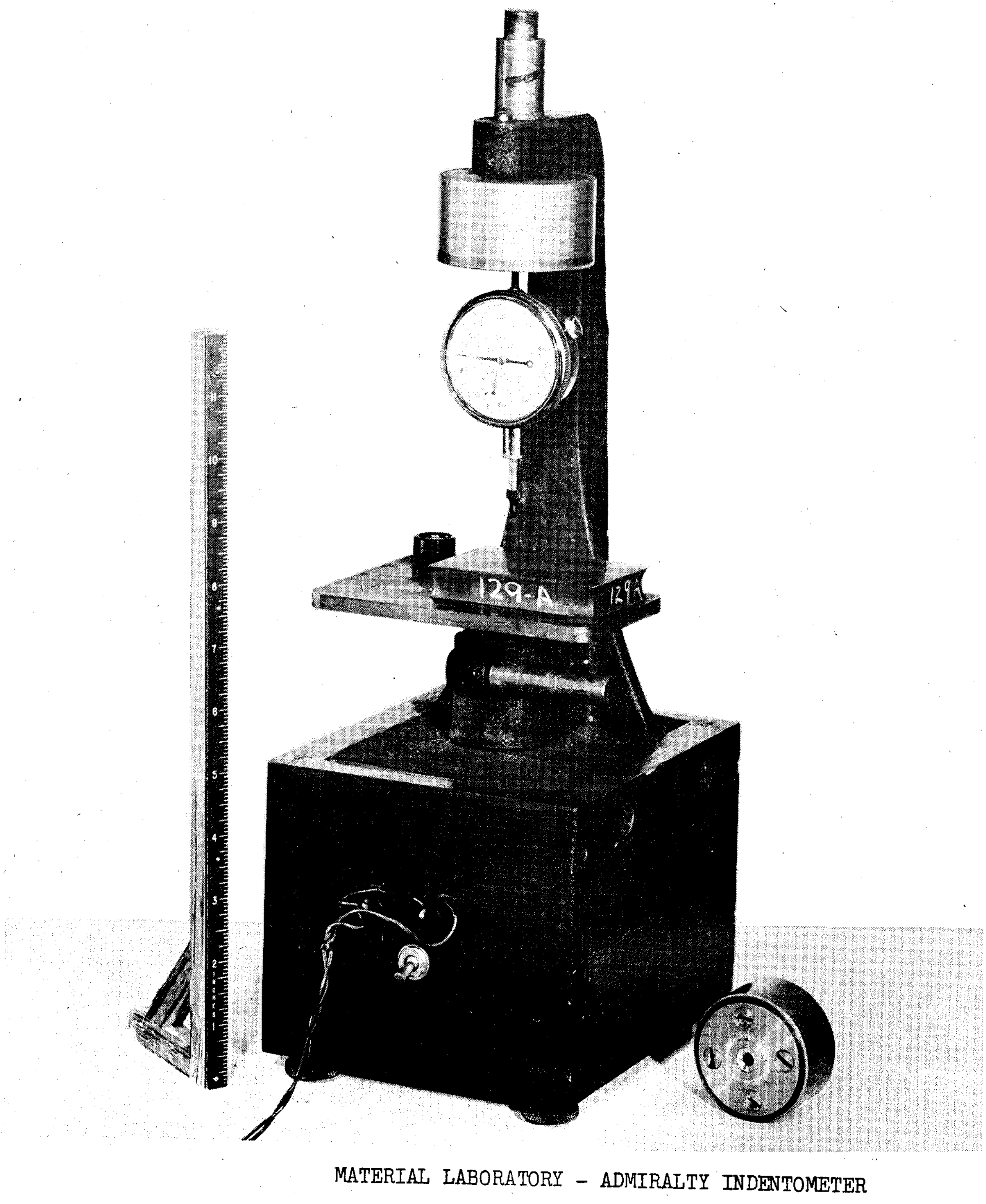
FIG. 1



MATERIAL LABORATORY - ADMIRALTY INDENTOMETER

NEW YORK NAVAL SHIPYARD

FIG. 2

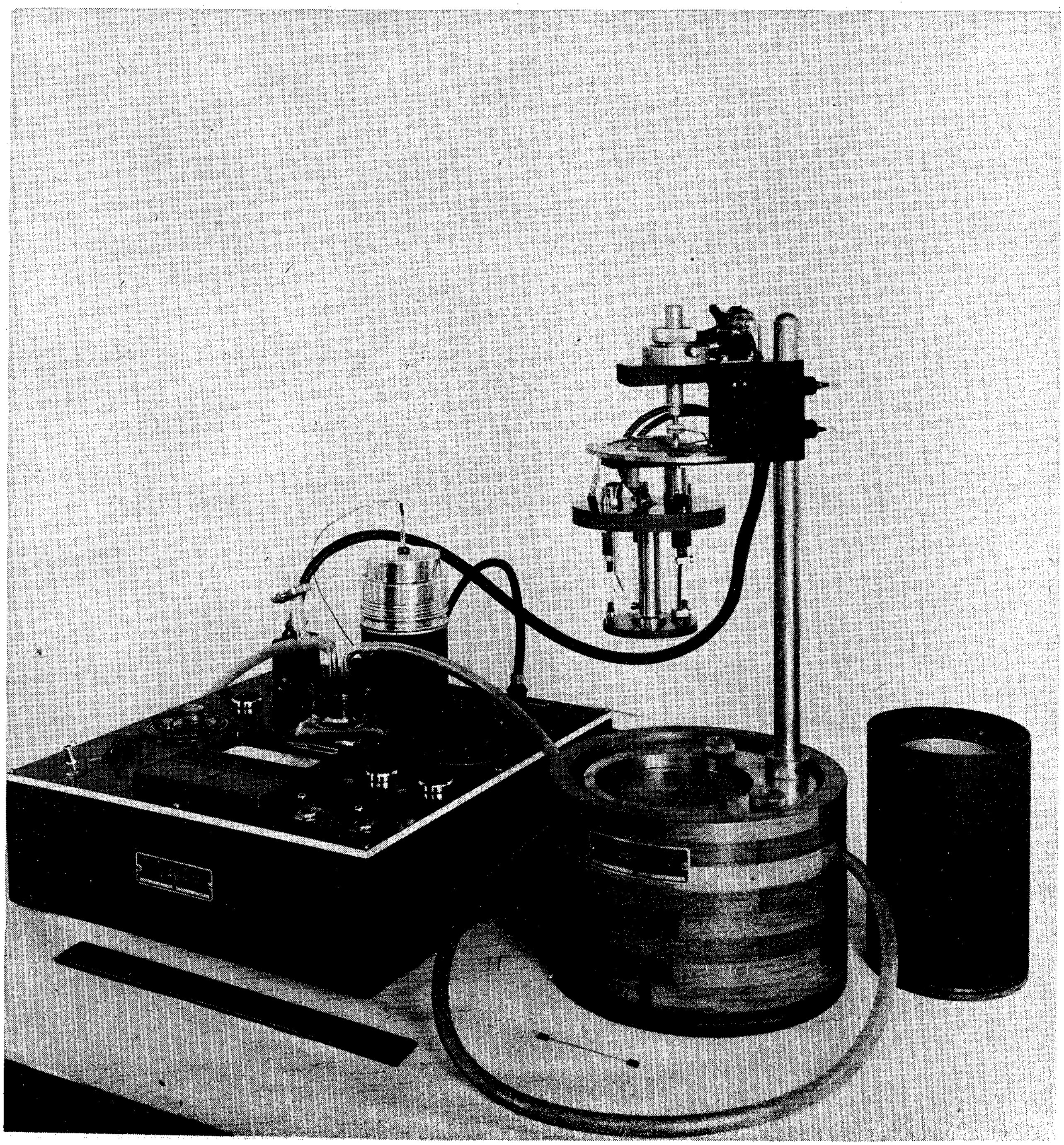


FIG. 3

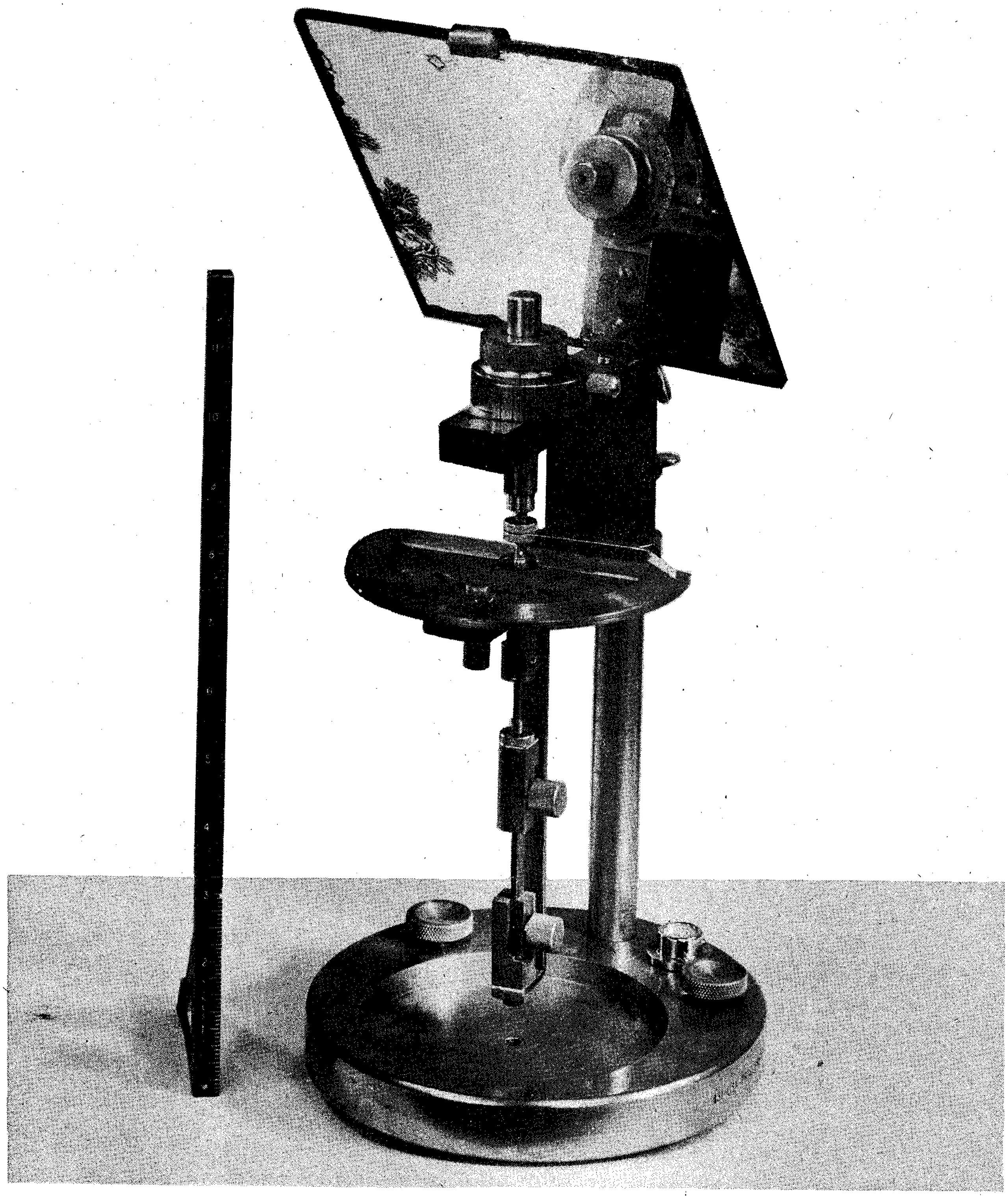


FIG. 4

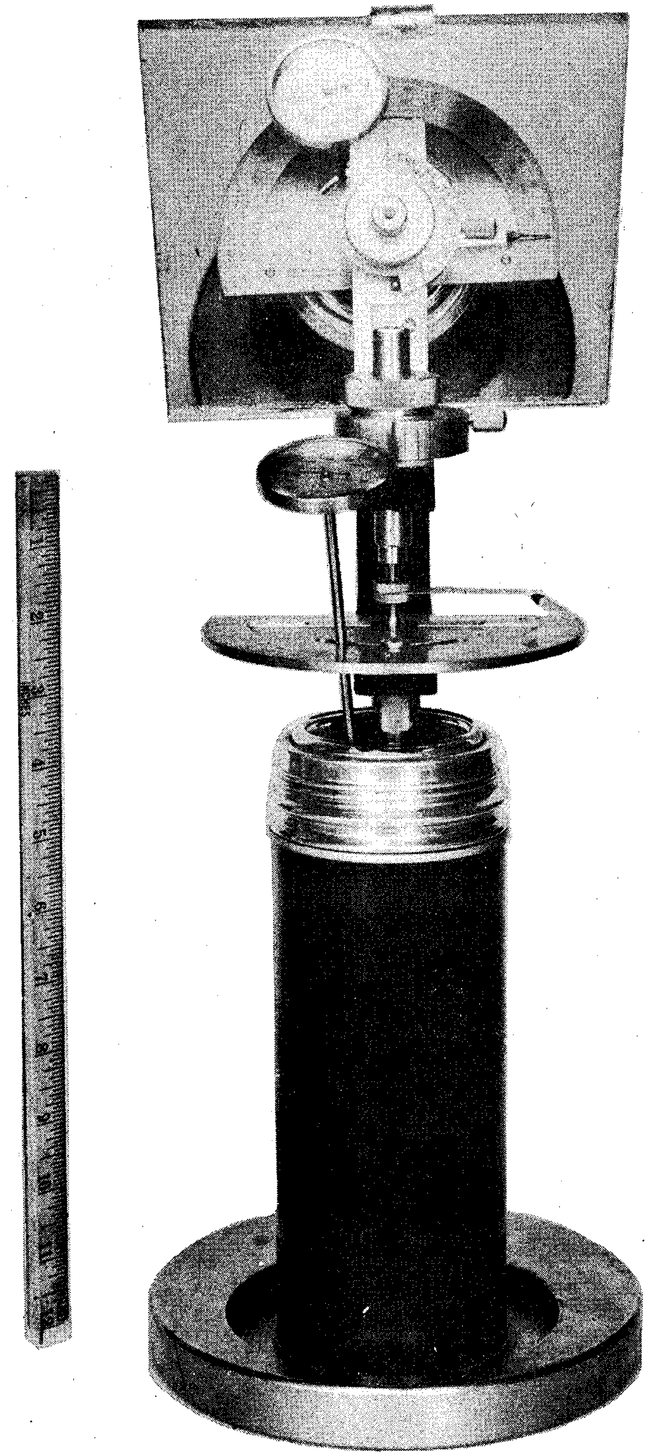


FIG. 5

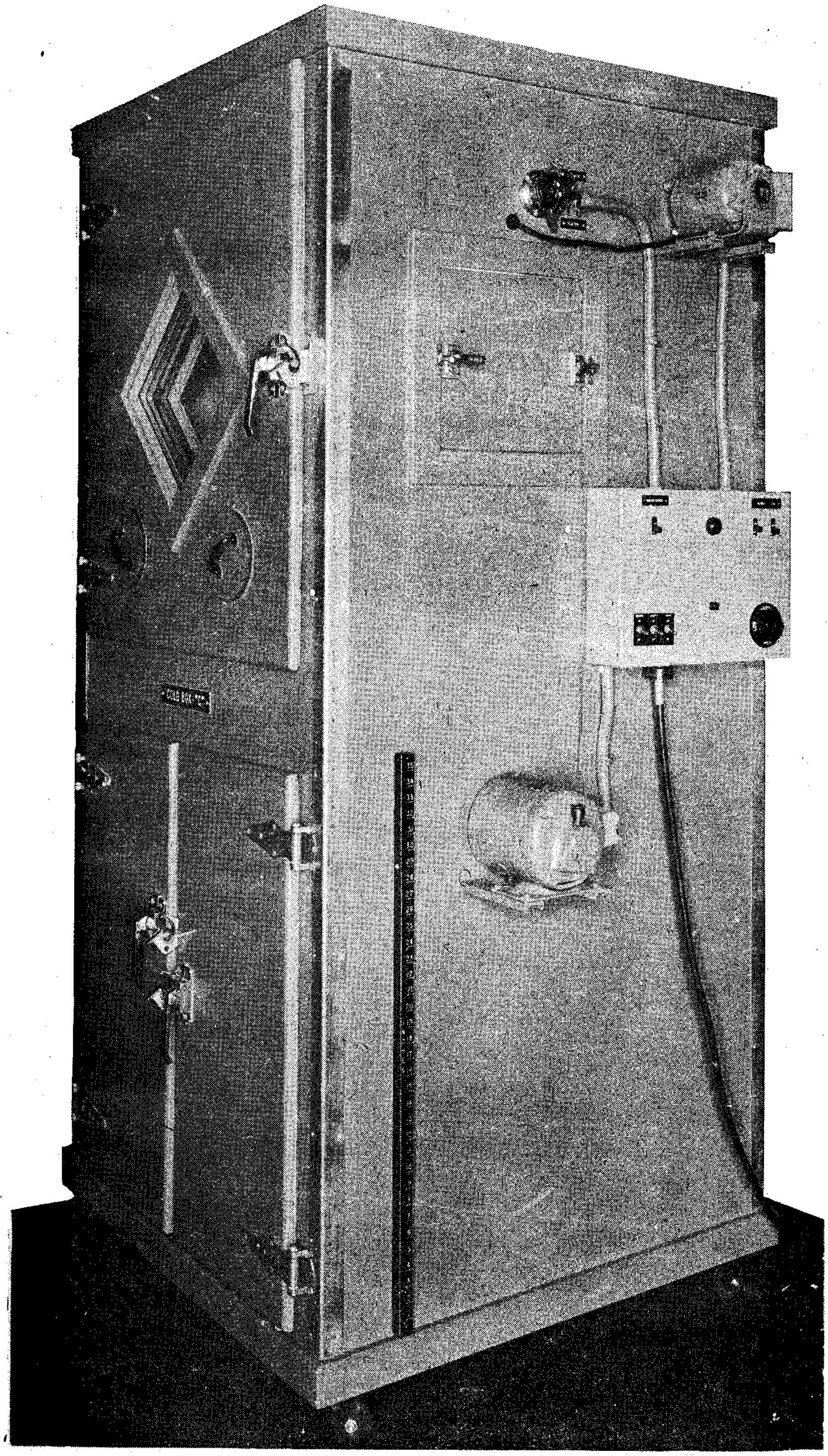


FIG. 6

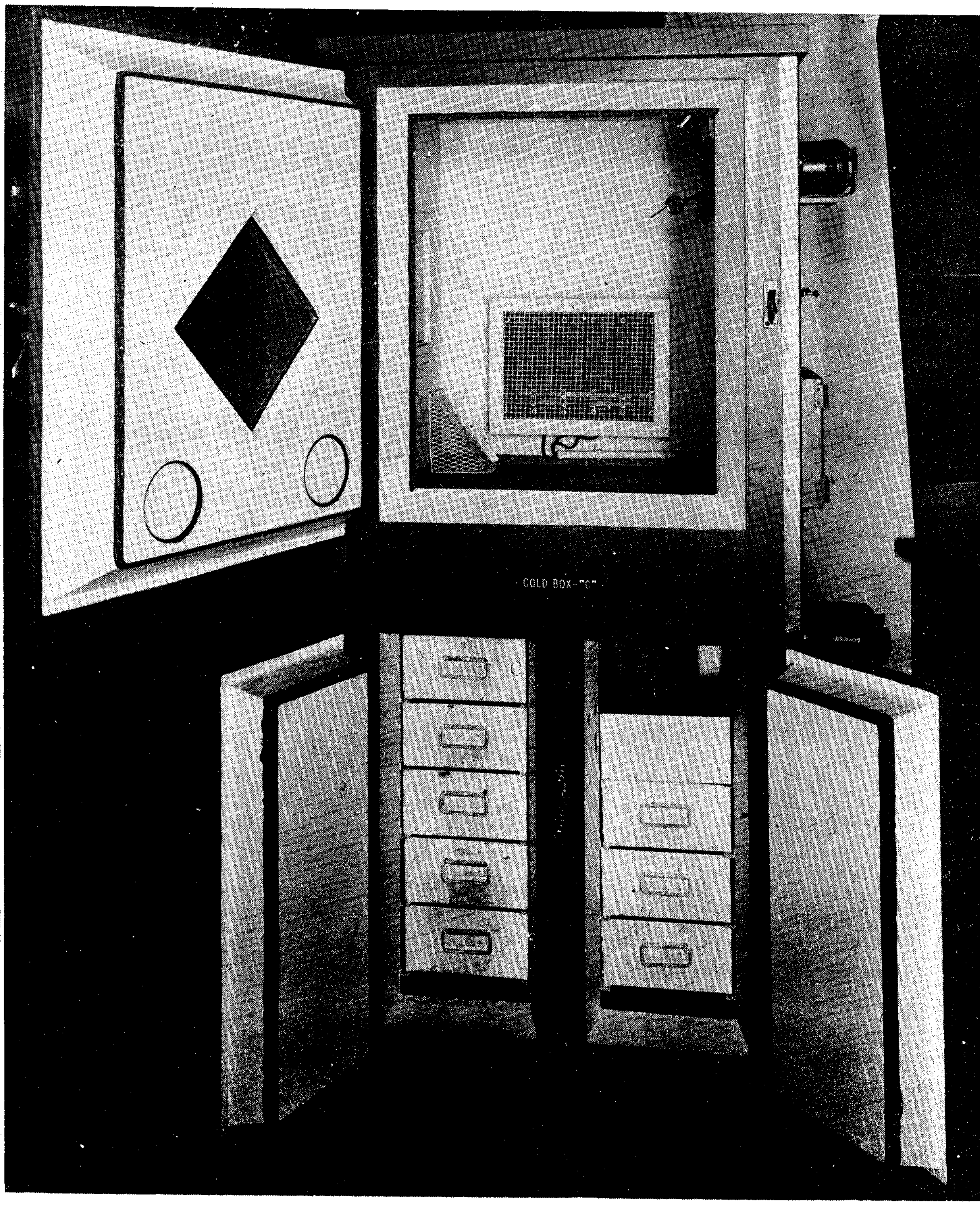


FIG. 7

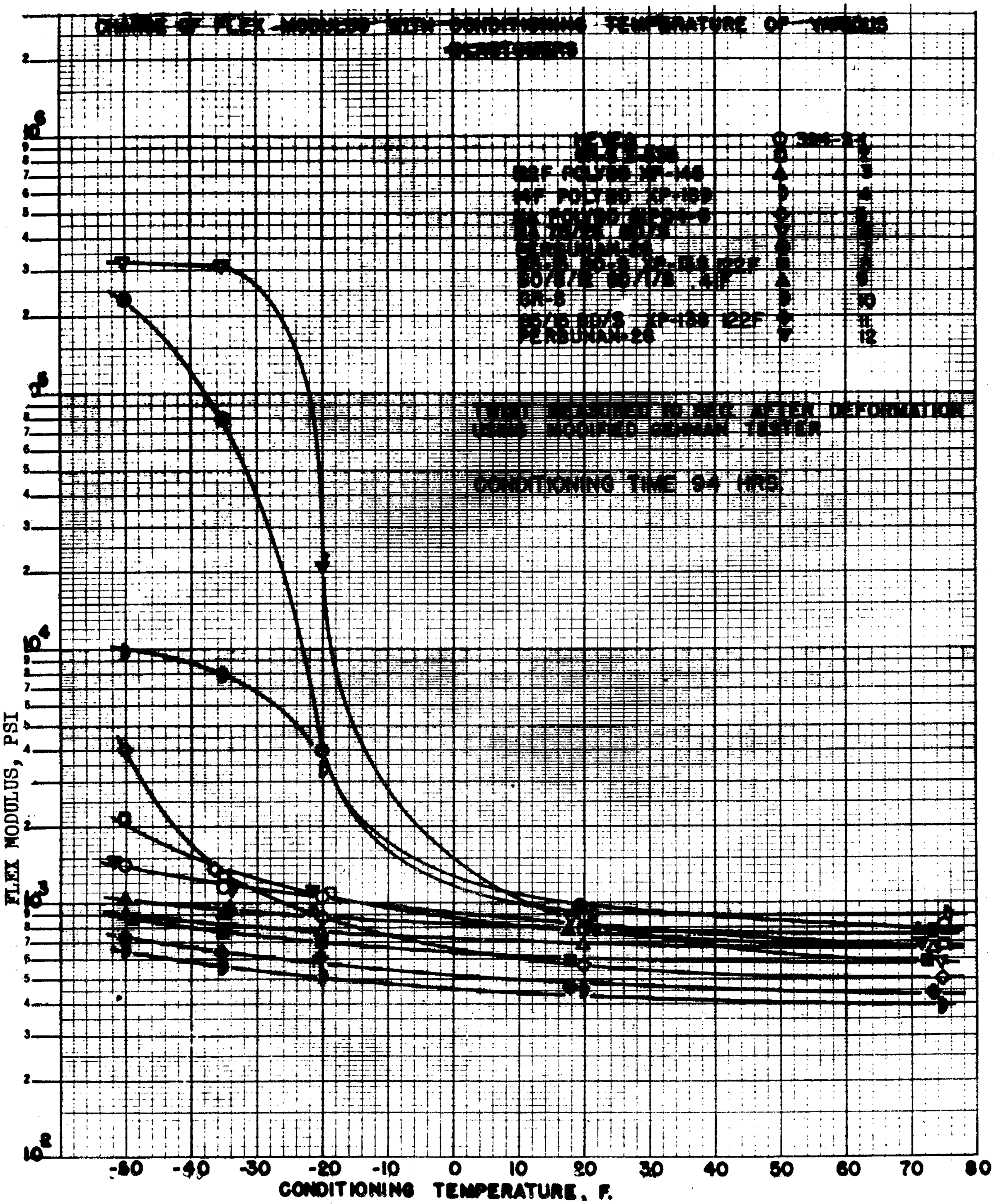
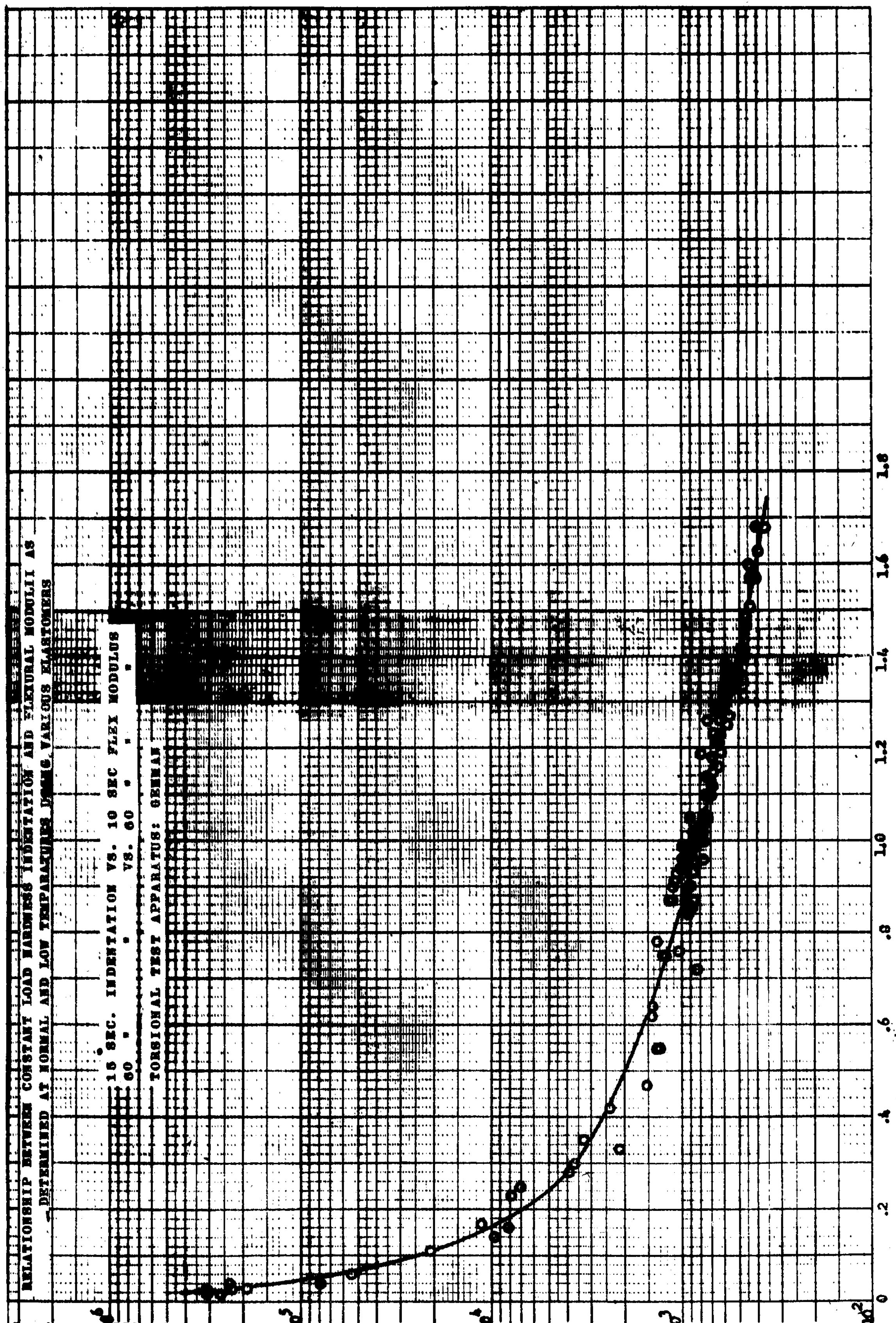
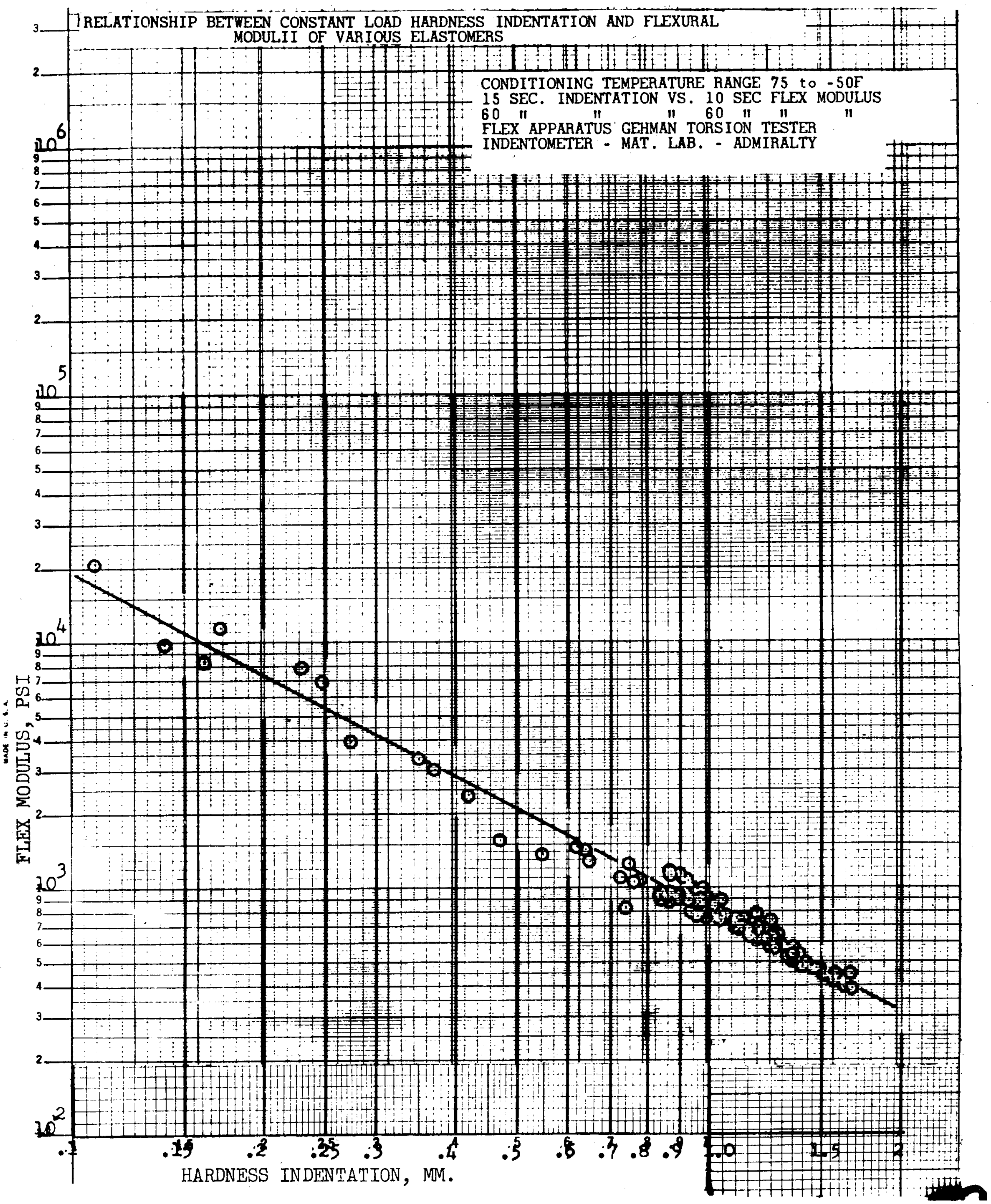

GR-S
PERBUNAN-26
CONDITIONING TIME 94 HRS.
FLEX MODULUS, PSI
10^6
10^5
10^4
10^3
10^2
-50 -40 -30 -20 -10 0 10 20 30 40 50 60 70 80
CONDITIONING TEMPERATURE, F.

FIG. 8



RELATIONSHIP BETWEEN CONSTANT LOAD HARDNESS INDENTATION AND FLEXURAL MODULII AS
DETERMINED AT NORMAL AND LOW TEMPARATURES
VARIOUS ELASTOMERS
15 SEC. INDENTATION VS. 10 SEC FLEX MODULUS
60 " VS. 60 " "
TORSIONAL TEST APPARATUS: GEHMAN
0
.2
.4
.6
.8
1.0
1.2
1.4
1.6
1.8
HARDNESS INDENTATION, MM
10^6
10^5
10^4
10^3
10^2
FLEX MODULUS, PSI

FIG. 9


RELATIONSHIP BETWEEN CONSTANT LOAD HARDNESS INDENTATION AND FLEXURAL MODULII OF VARIOUS ELASTOMERS
CONDITIONING TEMPERATURE RANGE 75 to -50F
15 SEC. INDENTATION VS. 10 SEC FLEX MODULUS
60 " " " 60 " " "
FLEX APPARATUS GEHMAN TORSION TESTER
INDENTOMETER - MAT. LAB. - ADMIRALTY
FLEX MODULUS, PSI
HARDNESS INDENTATION, MM.

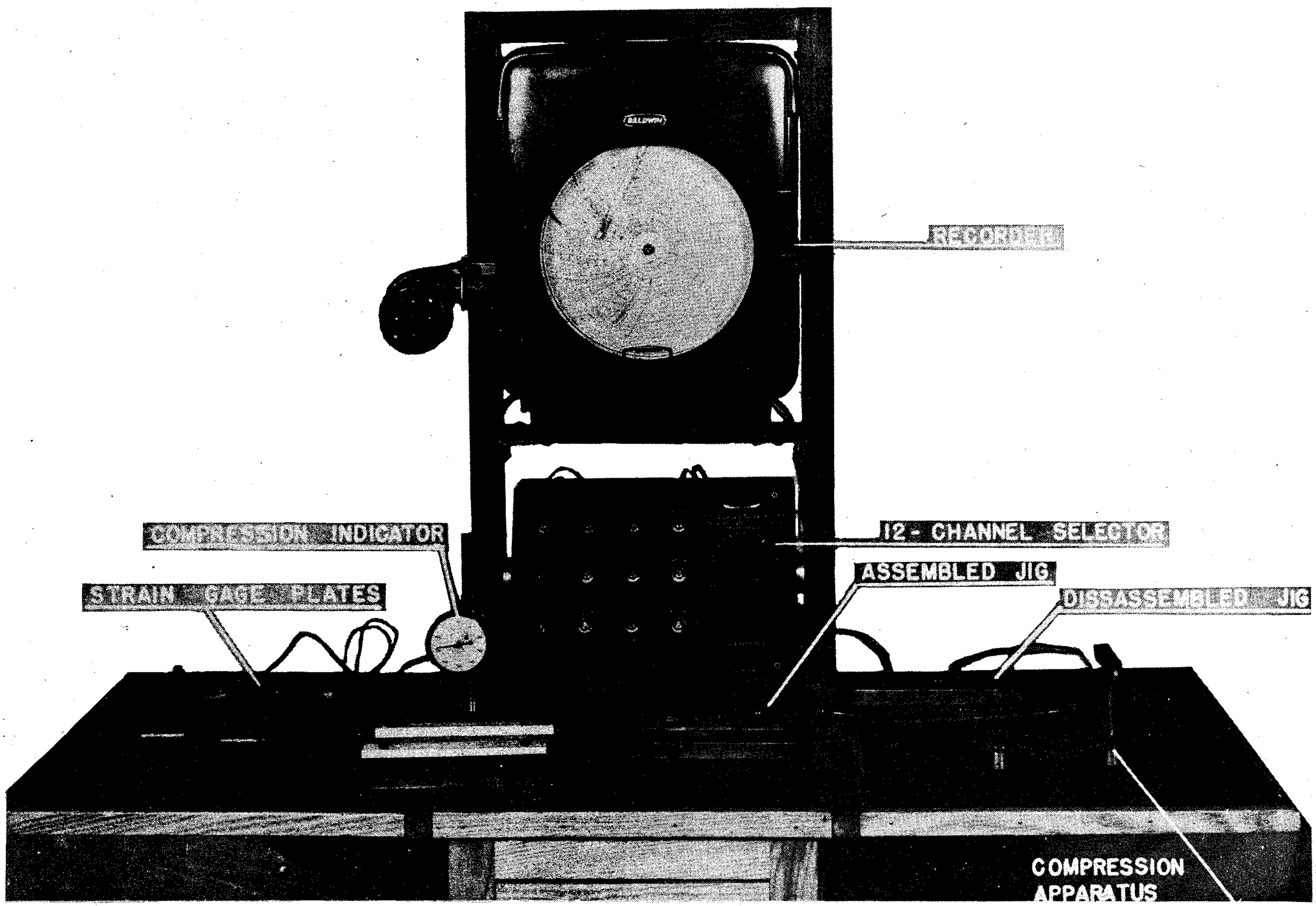
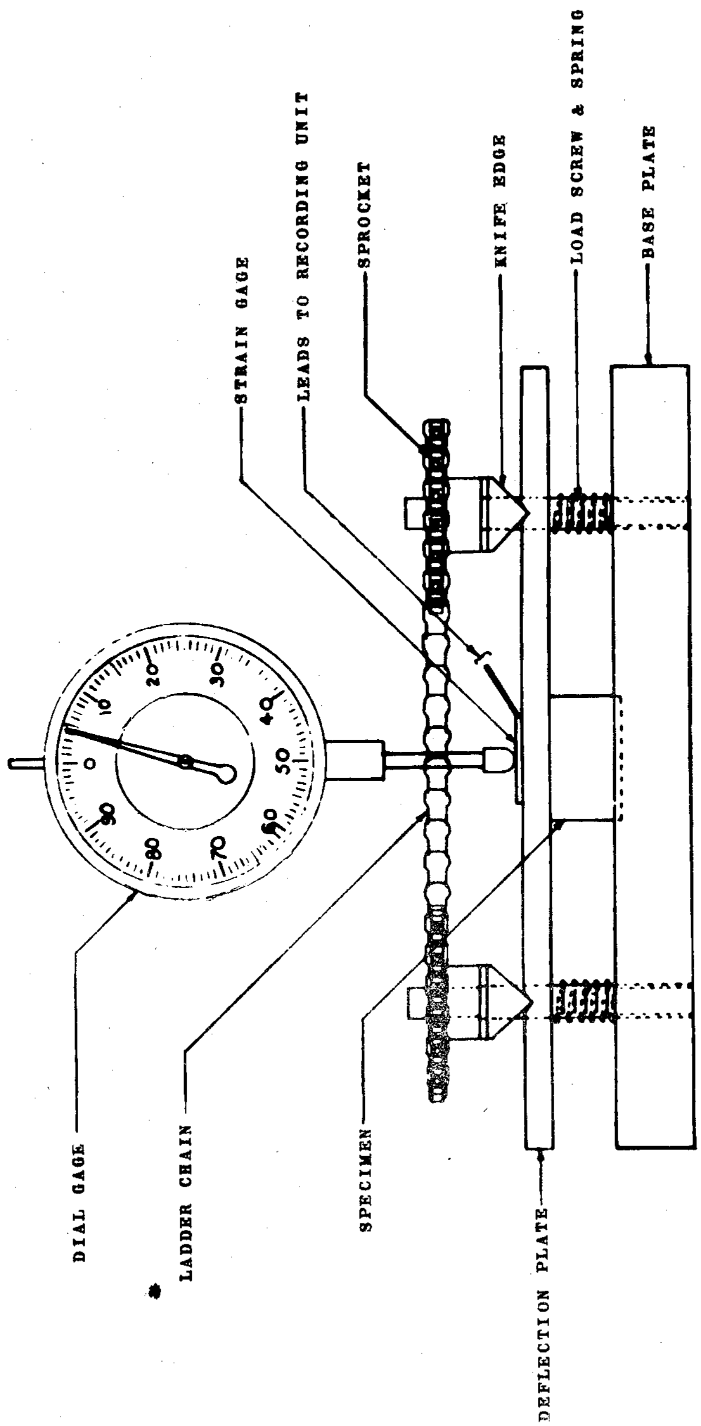

RECORDER
COMPRESSION INDICATOR
STRAIN GAGE PLATES
12- CHANNEL SELECTOR
ASSEMBLED JIG
DISSASSEMBLED JIG
COMPRESSION APPARATUS

FIG. 10

FIG. 11



STRAIN-GAGE STRESS DECAY APPARATUS

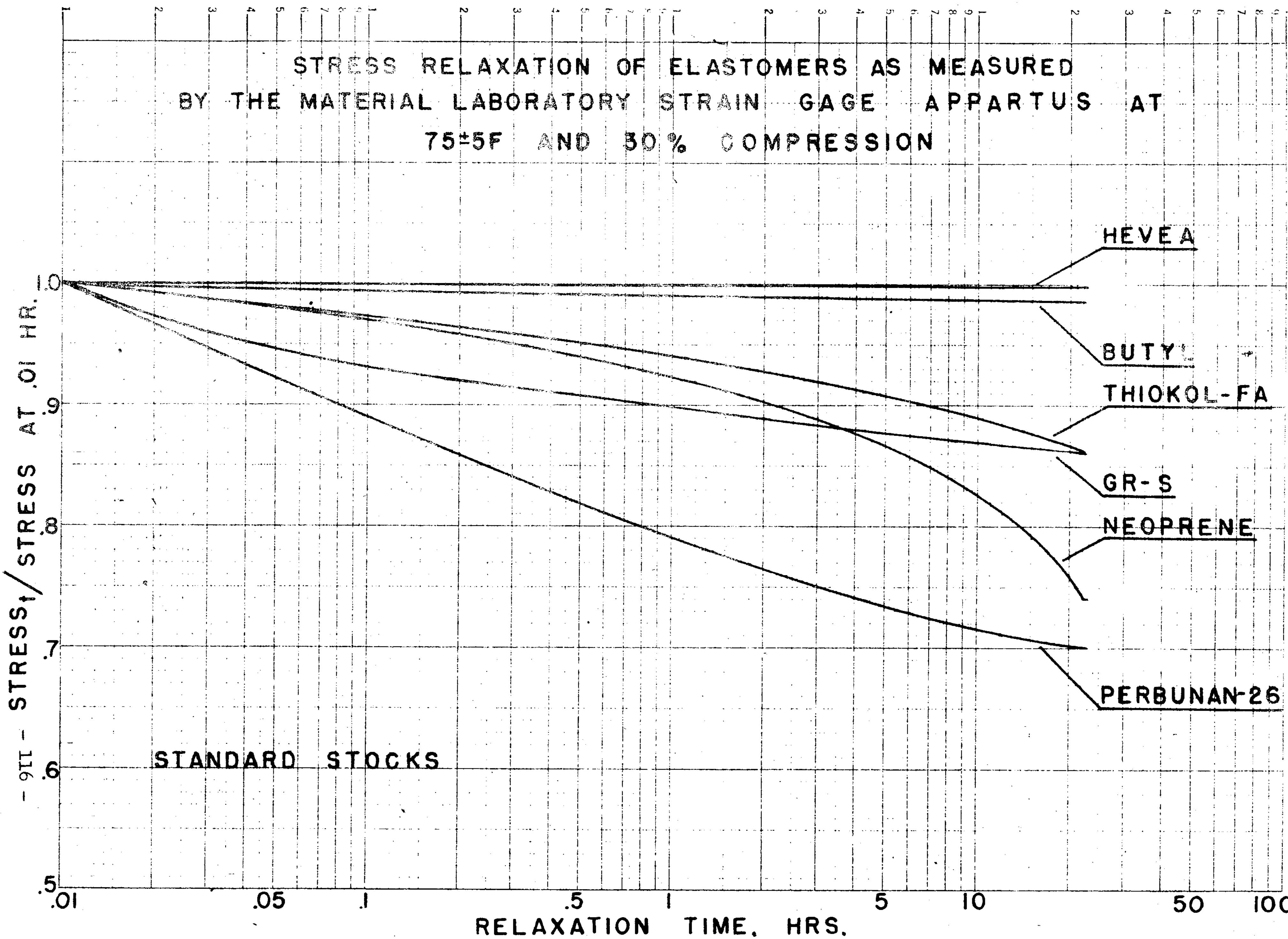

STRESS RELAXATION OF ELASTOMERS AS MEASURED
BY THE MATERIAL LABORATORY STRAIN GAGE APPARTUS AT
75±5F AND 30% COMPRESSION
HEVEA
BUTYL
THIOKOL-FA
GR-S
NEOPRENE
PERBUNAN-26
STANDARD STOCKS
STRESS$_t$/STRESS AT .01 HR.
1.0
.9
.8
.7
.6
.5
.01
.05
.1
.5
1
5
10
50
100
RELAXATION TIME. HRS.

FIG. 12

FIG. 13

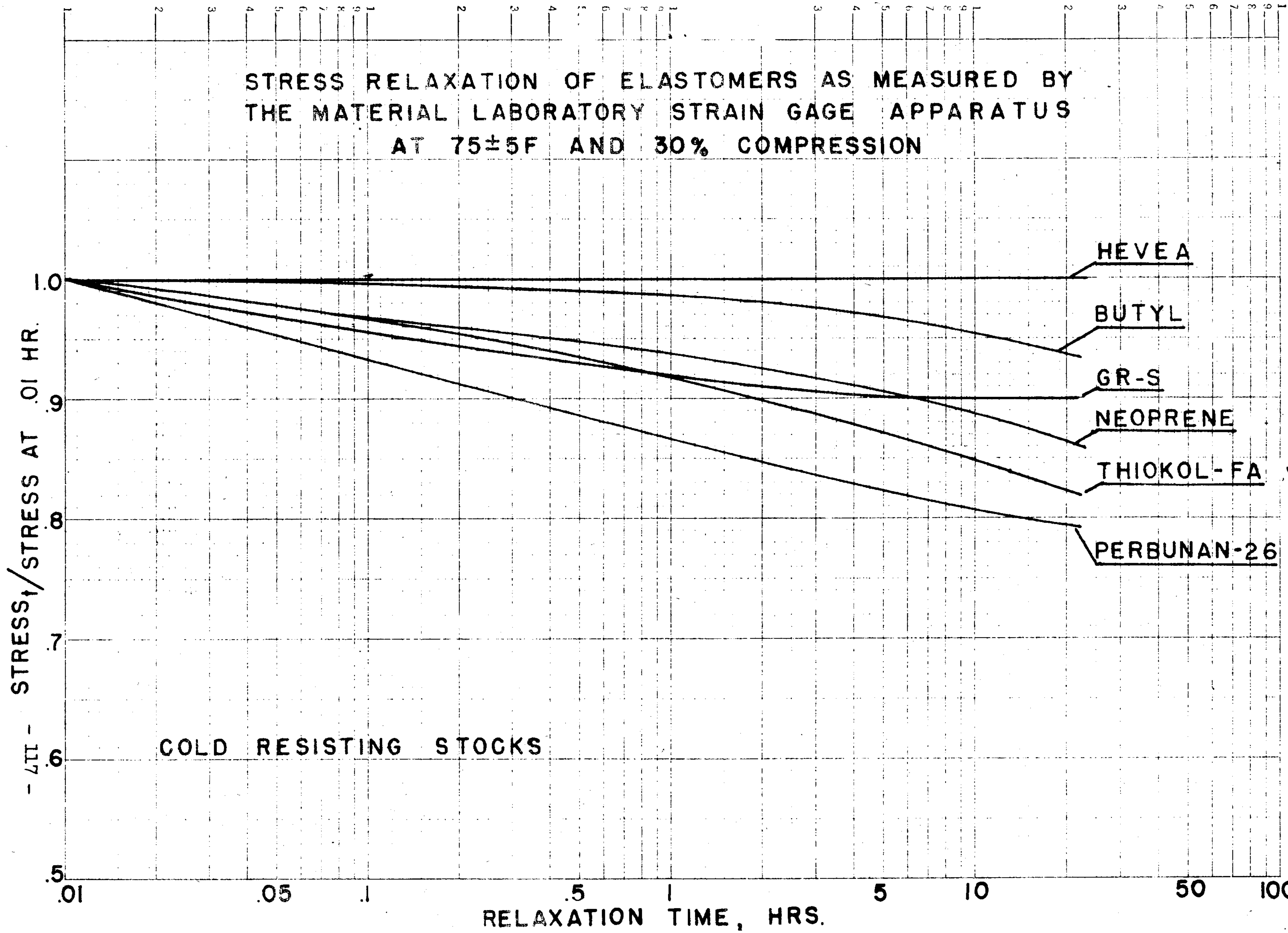

STRESS RELAXATION OF ELASTOMERS AS MEASURED BY
THE MATERIAL LABORATORY STRAIN GAGE APPARATUS
AT 75±5F AND 30% COMPRESSION
HEVEA
BUTYL
GR-S
NEOPRENE
THIOKOL-FA
PERBUNAN-26
COLD RESISTING STOCKS
STRESS$_t$/STRESS AT .01 HR.
1.0
.9
.8
.7
.6
.5
.01
.05
.1
.5
1
5
10
50
100
RELAXATION TIME, HRS.

FIG. 14

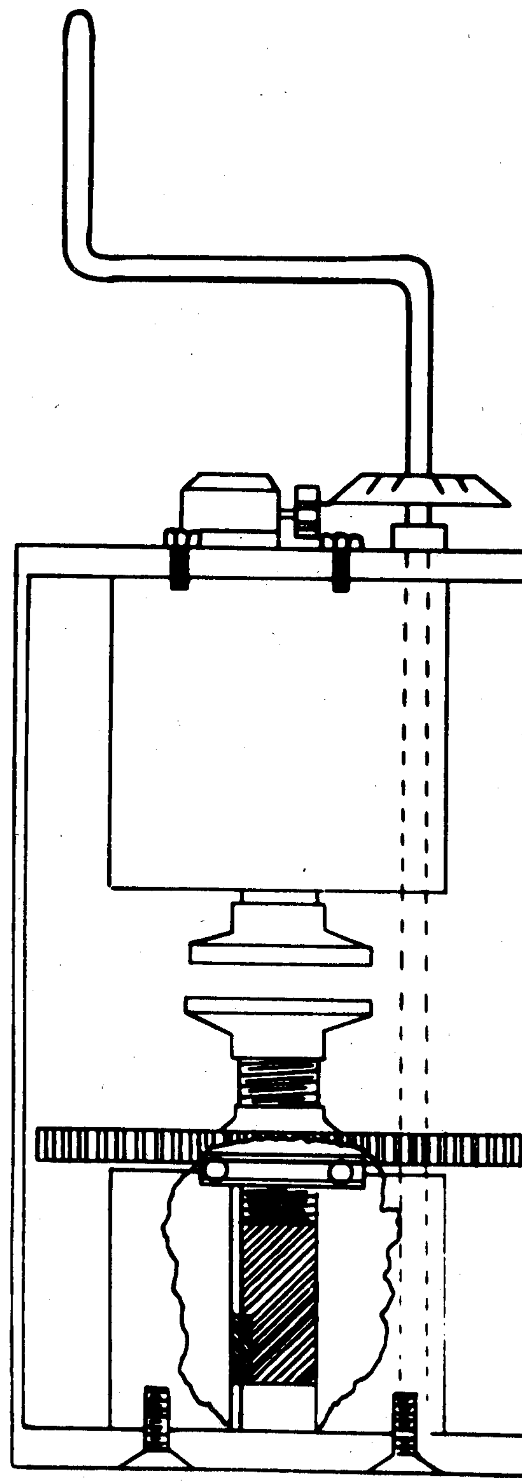


FIG. 15

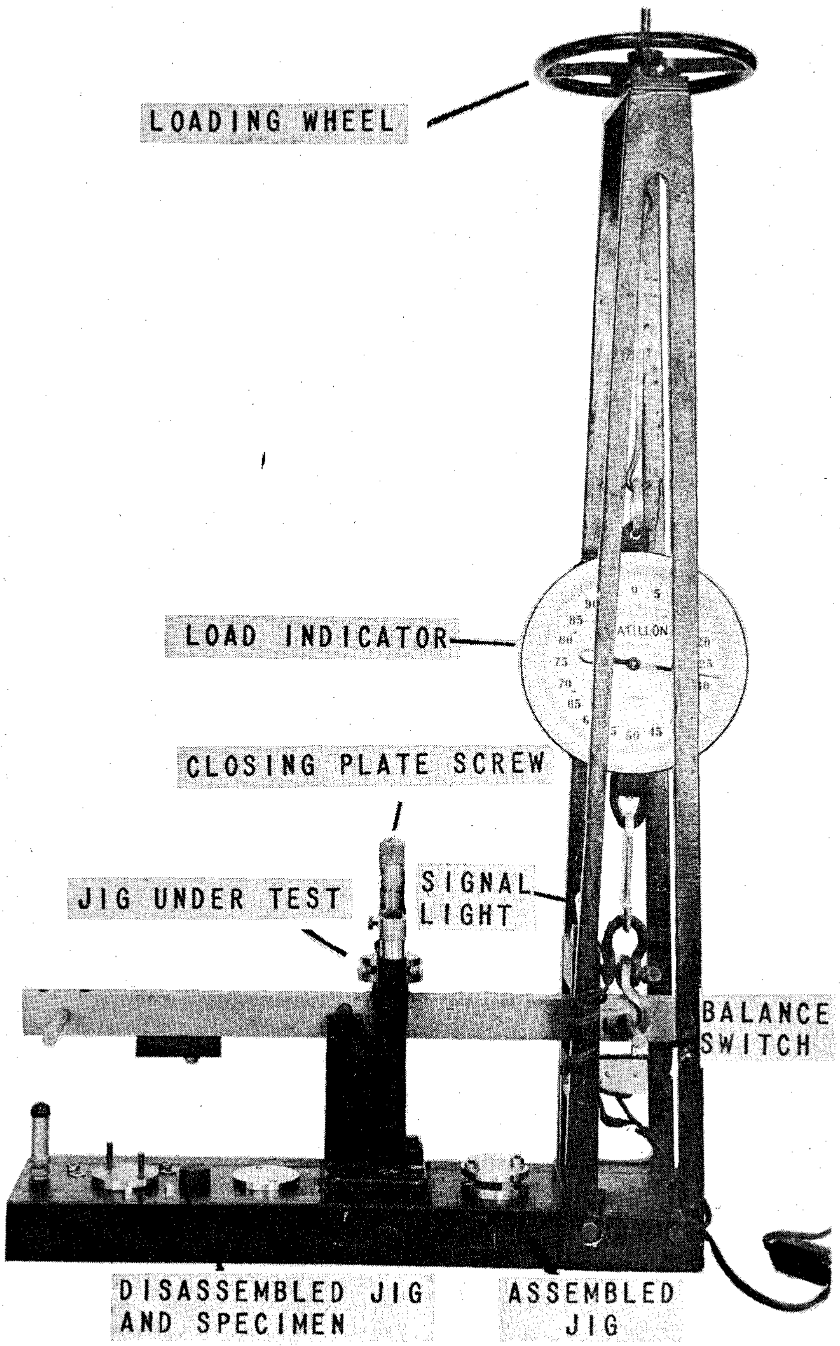
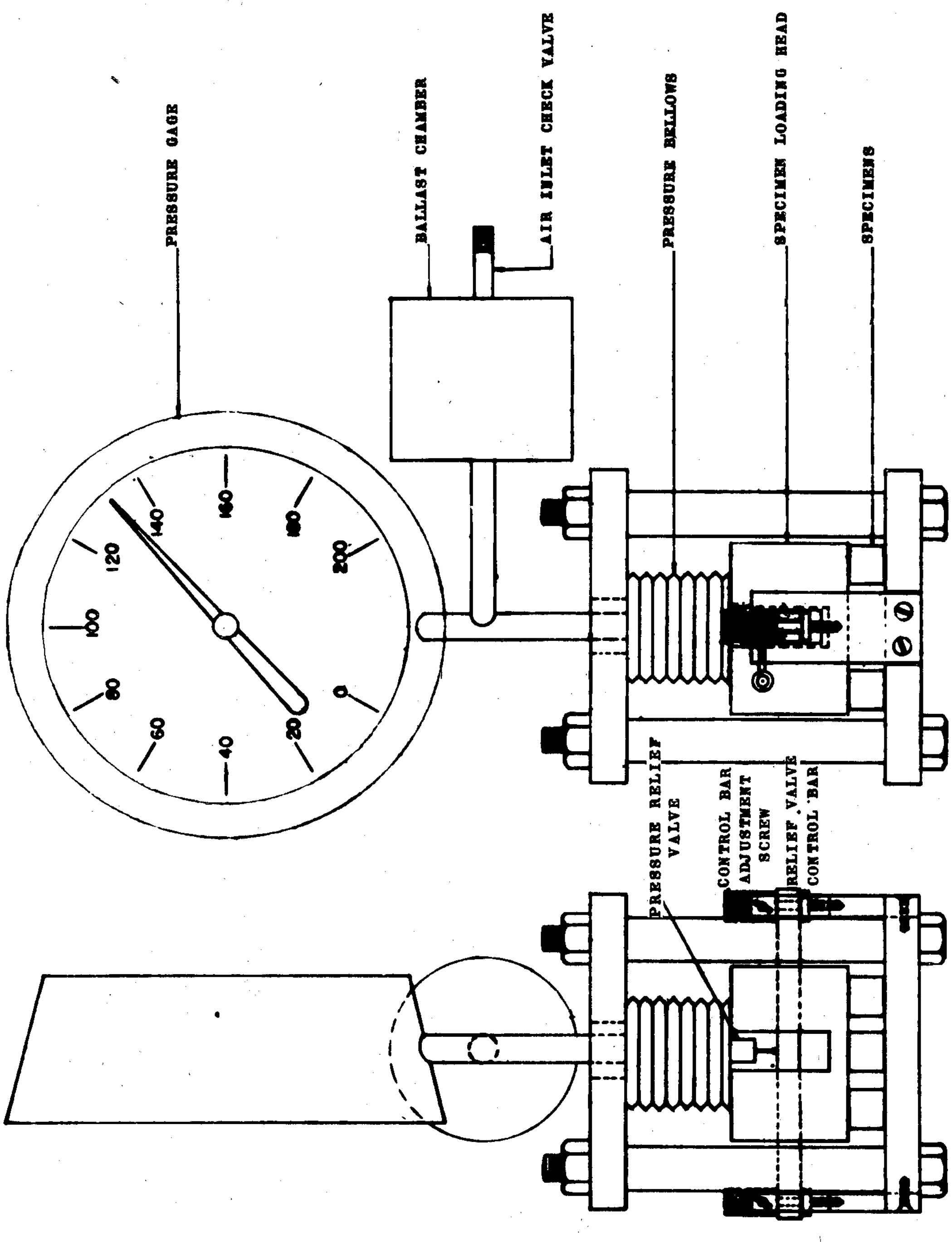

LOADING WHEEL
LOAD INDICATOR
CLOSING PLATE SCREW
SIGNAL LIGHT
JIG UNDER TEST
BALANCE SWITCH
DISASSEMBLED JIG AND SPECIMEN
ASSEMBLED JIG

FIG. 16



GEHMAN STRESS RELAXATION APPARATUS

FIG. 17

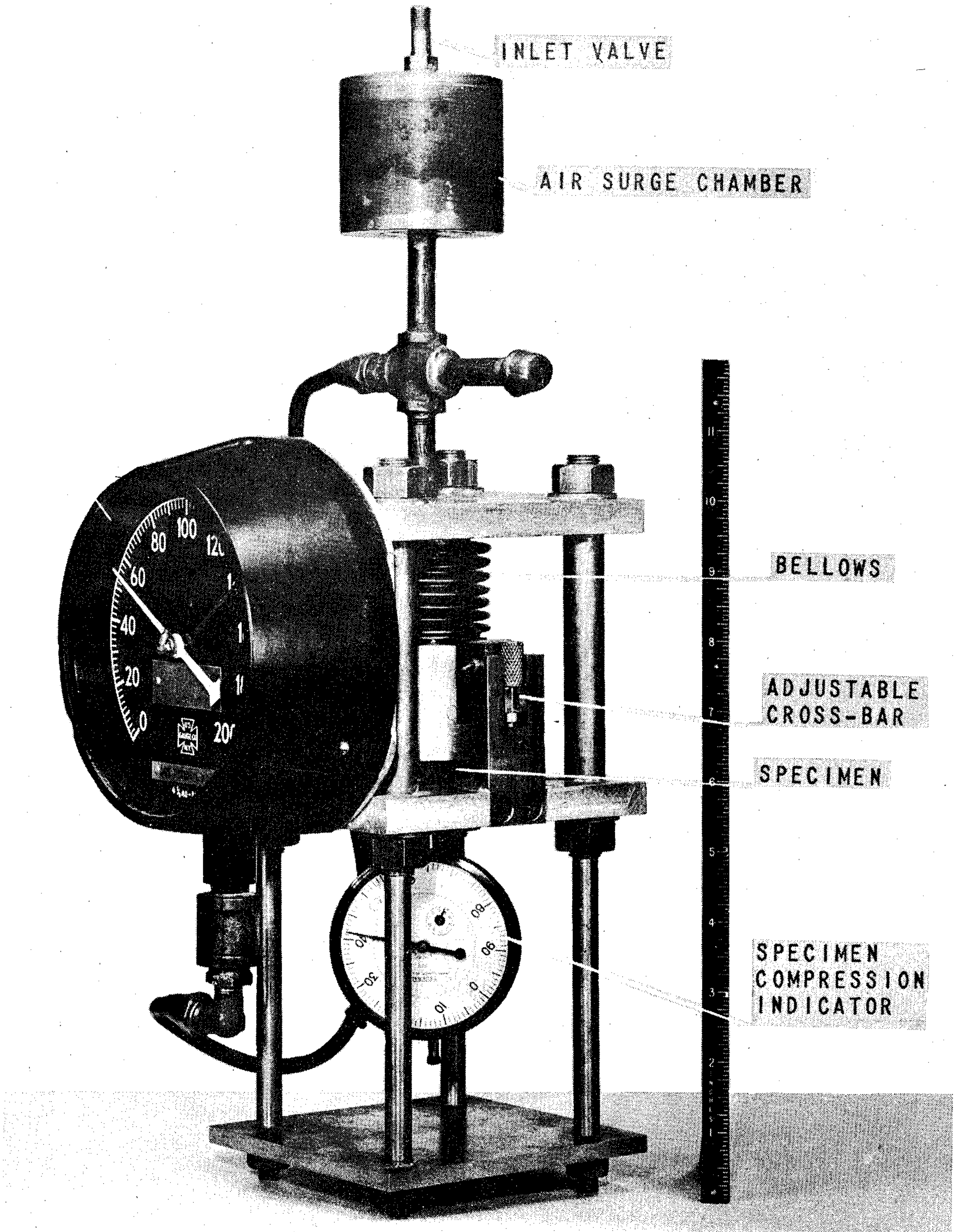

INLET VALVE
AIR SURGE CHAMBER
BELLOWS
ADJUSTABLE CROSS-BAR
SPECIMEN
SPECIMEN COMPRESSION INDICATOR

FIG. 18

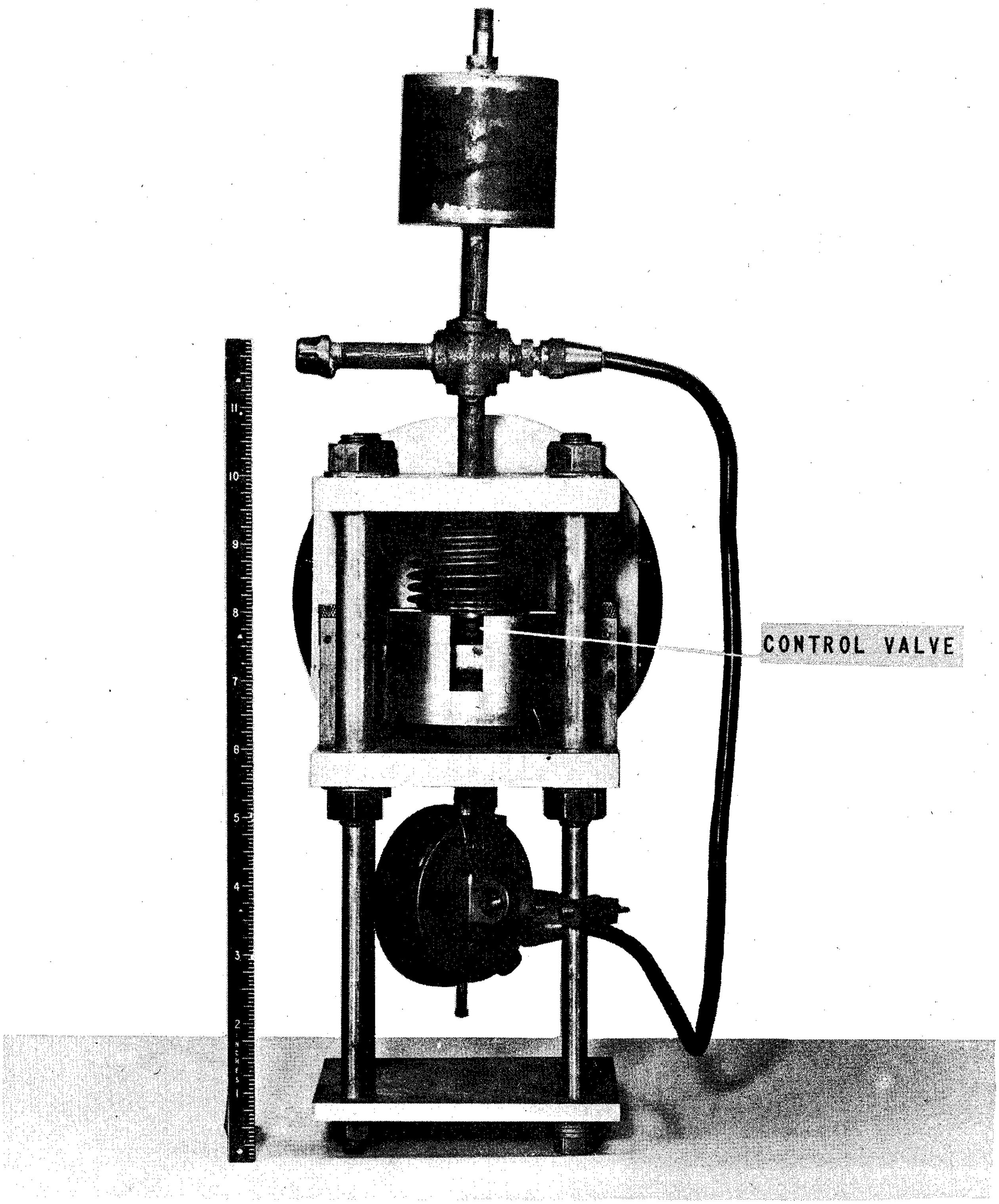


FIG. 19

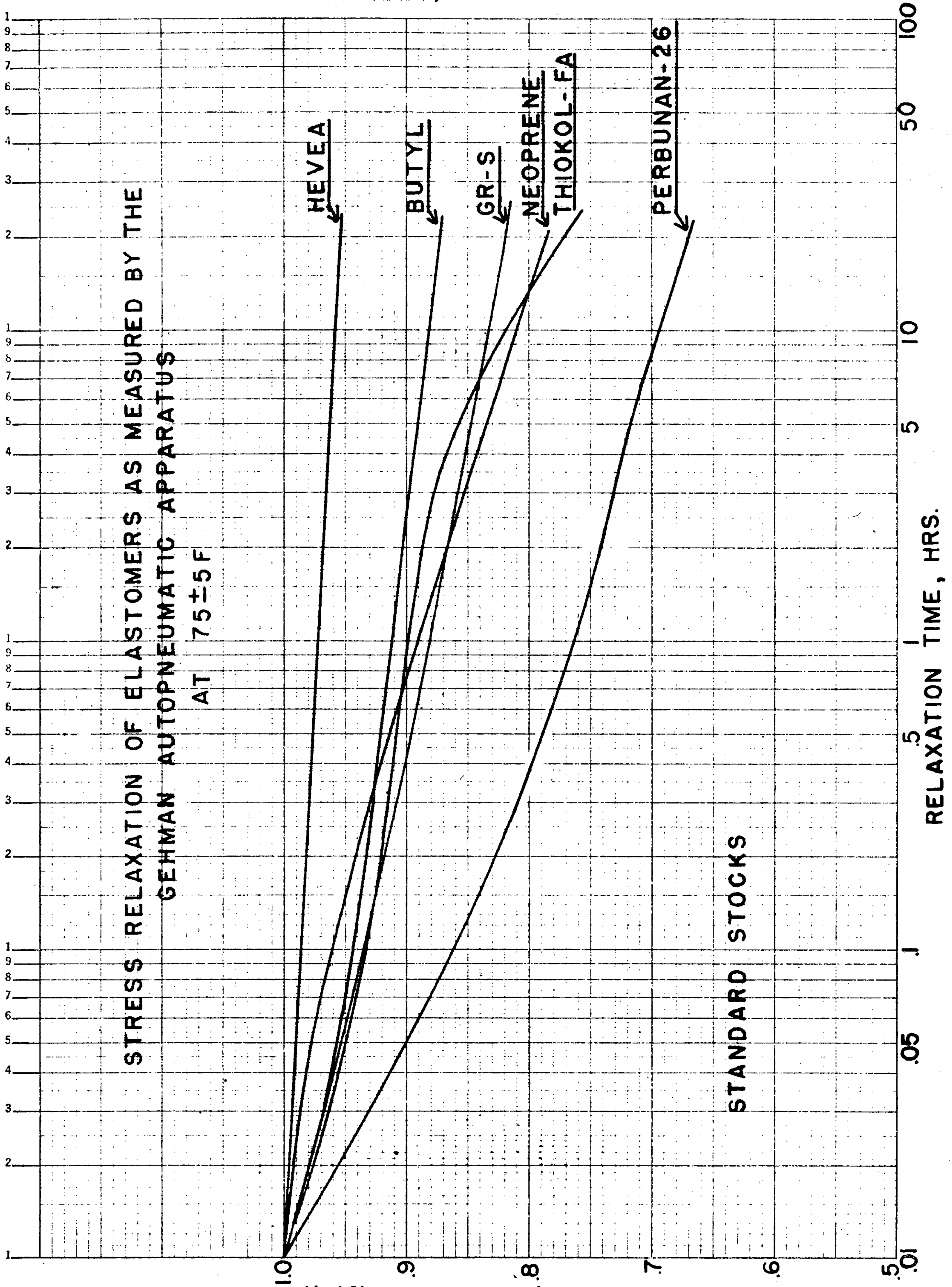
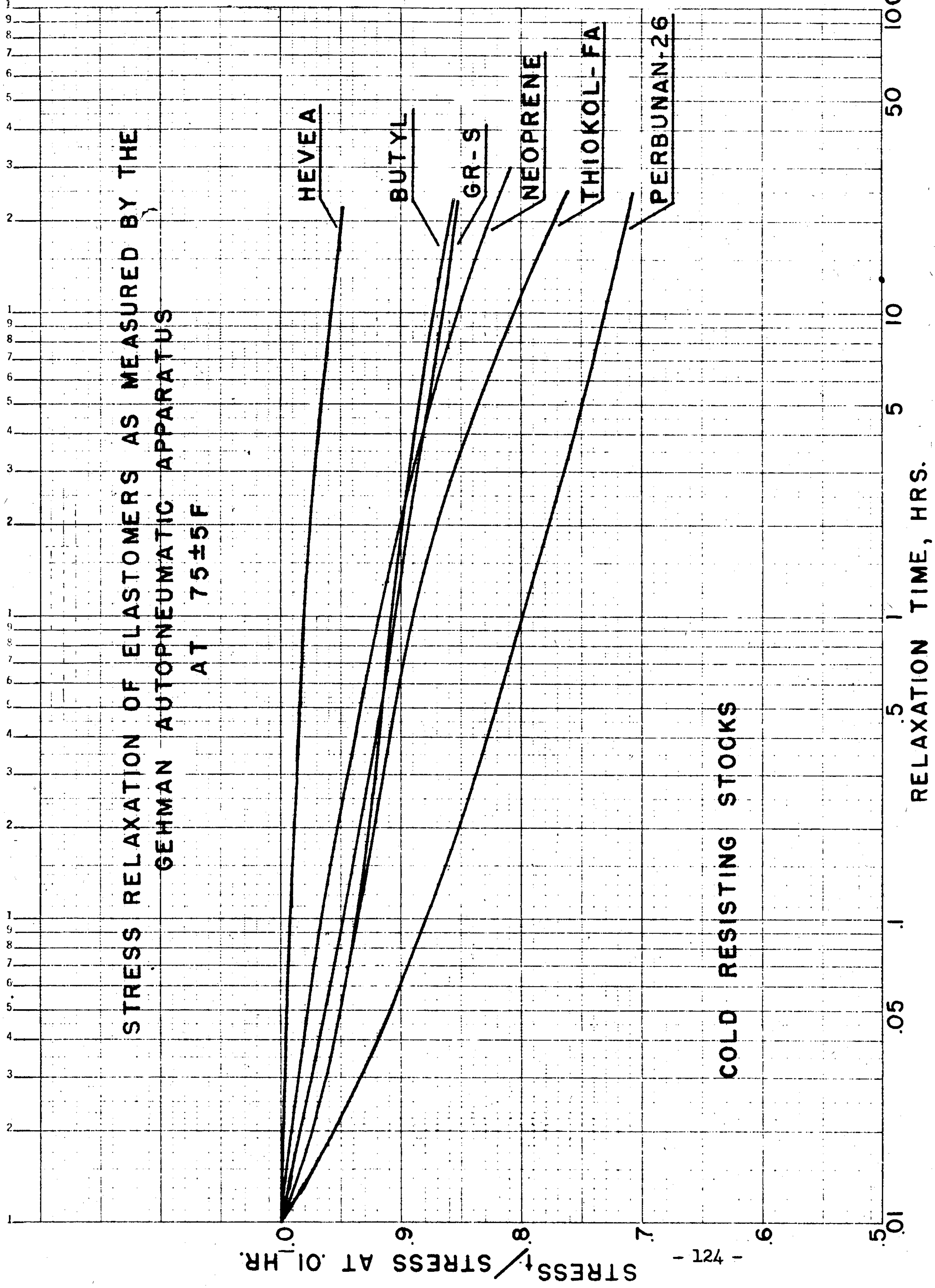

STRESS RELAXATION OF ELASTOMERS AS MEASURED BY THE
GEHMAN AUTOPNEUMATIC APPARATUS
AT 75±5F
STANDARD STOCKS
HEVEA
BUTYL
GR-S
NEOPRENE
THIOKOL-FA
PERBUNAN-26
RELAXATION TIME, HRS.
.01
.05
.1
.5
1
5
10
50
100
1.0
.9
.8
.7
.6
.5
STRESS$_t$/STRESS AT .01 HR.

FIG. 20



STRESS RELAXATION OF ELASTOMERS AS MEASURED BY THE
GEHMAN AUTOPNEUMATIC APPARATUS
AT 75±5F
COLD RESISTING STOCKS
HEVEA
BUTYL
GR-S
NEOPRENE
THIOKOL-FA
PERBUNAN-26
RELAXATION TIME, HRS.
.01
.05
.1
.5
1
5
10
50
100
1.0
.9
.8
.7
.6
.5
STRESS$_t$/STRESS AT .01 HR.

FIG. 21

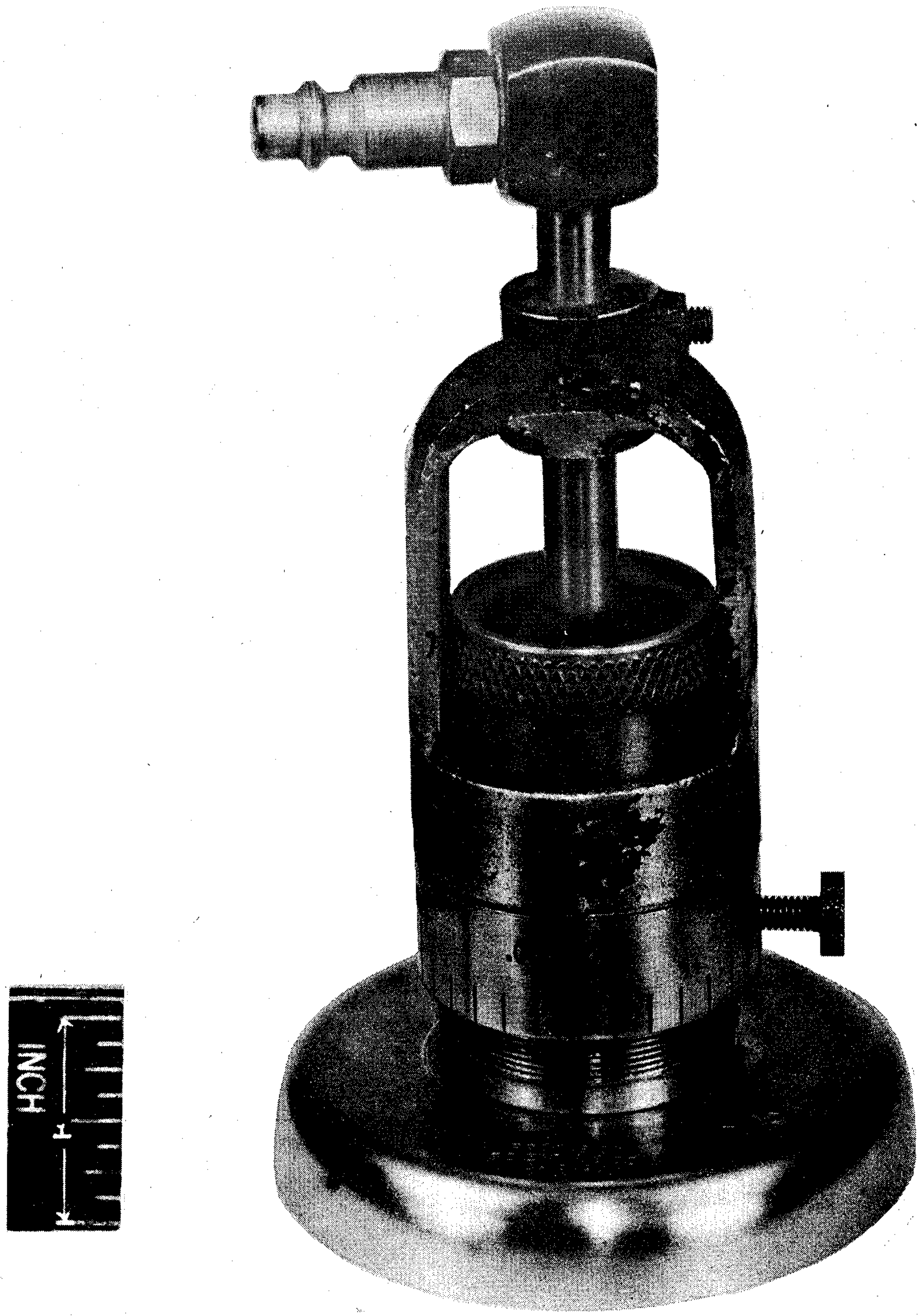
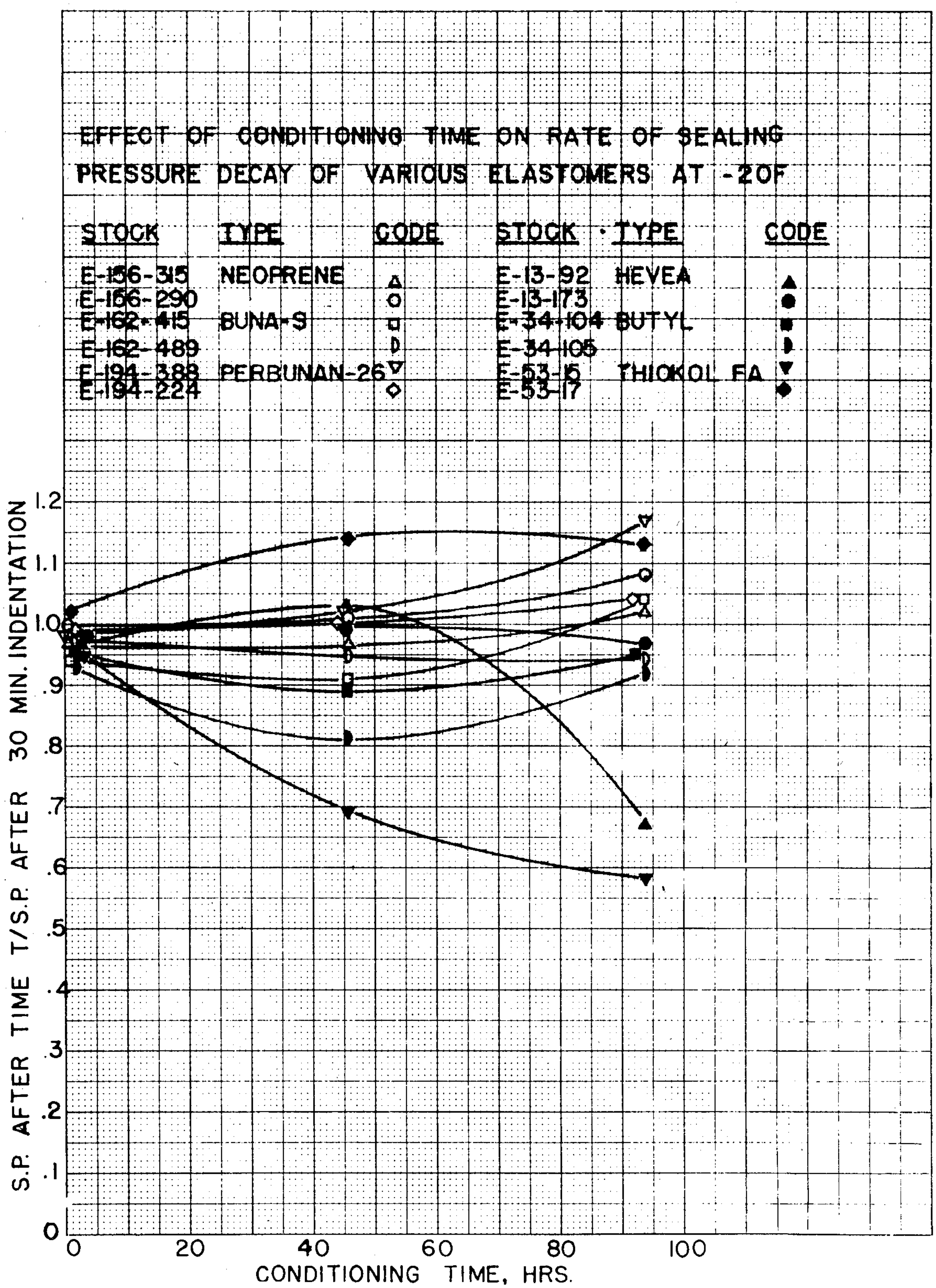
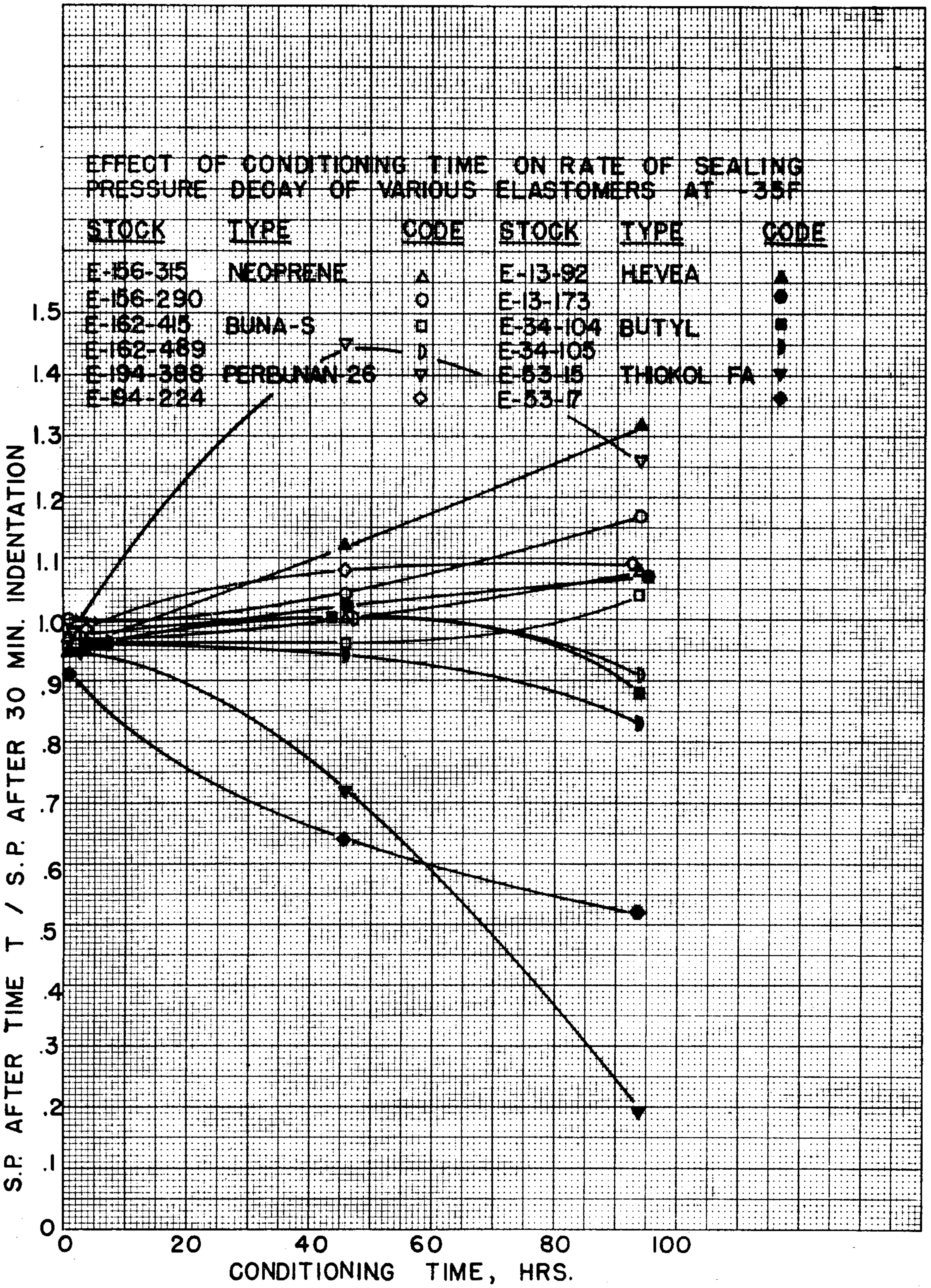


FIG. 22



EFFECT OF CONDITIONING TIME ON RATE OF SEALING PRESSURE DECAY OF VARIOUS ELASTOMERS AT -20F
STOCK TYPE CODE STOCK TYPE CODE
E-156-315 NEOPRENE △ E-13-92 HEVEA ▲
E-156-290 ○ E-13-173 ●
E-162-415 BUNA-S □ E-34-104 BUTYL ■
E-162-489 ◗ E-34-105 ◗
E-194-388 PERBUNAN-26 ▽ E-53-15 THIOKOL FA ▼
E-194-224 ◇ E-53-17 ◆
S.P. AFTER TIME T/S.P. AFTER 30 MIN. INDENTATION
0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1.0
1.1
1.2
0
20
40
60
80
100
CONDITIONING TIME, HRS.

FIG. 23



EFFECT OF CONDITIONING TIME ON RATE OF SEALING PRESSURE DECAY OF VARIOUS ELASTOMERS AT -35F
STOCK TYPE CODE STOCK TYPE CODE
E-156-315 NEOPRENE △ E-13-92 HEVEA ▲
E-156-290 ○ E-13-173 ●
E-162-415 BUNA-S □ E-34-104 BUTYL ■
E-162-489 E-34-105
E-194-388 PERBUNAN 26 ▽ E-53-15 THIOKOL FA ▼
E-194-224 ◇ E-53-17 ◆
S.P. AFTER TIME T / S.P. AFTER 30 MIN. INDENTATION
0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1.0
1.1
1.2
1.3
1.4
1.5
0
20
40
60
80
100
CONDITIONING TIME, HRS.

FIG. 24

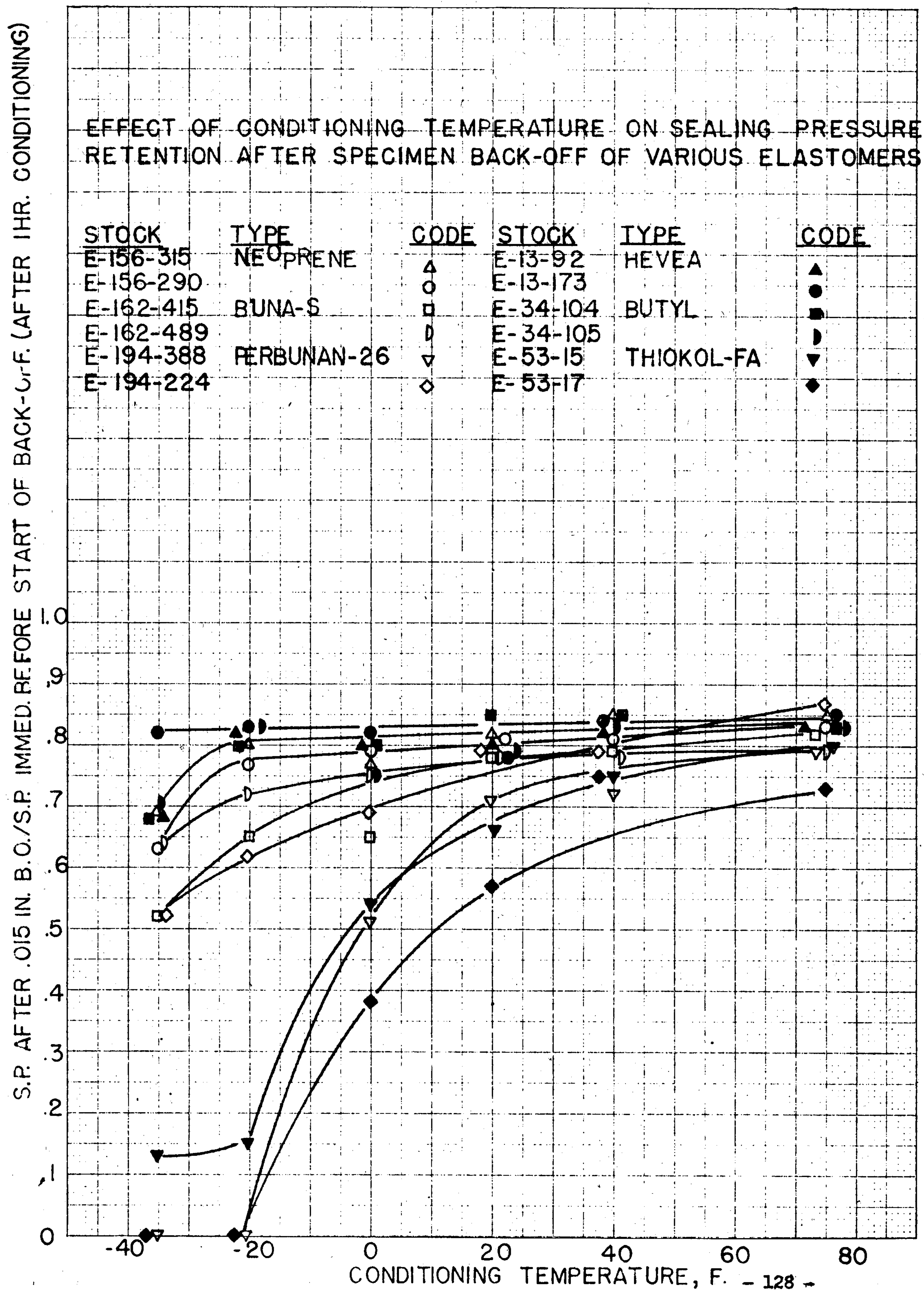
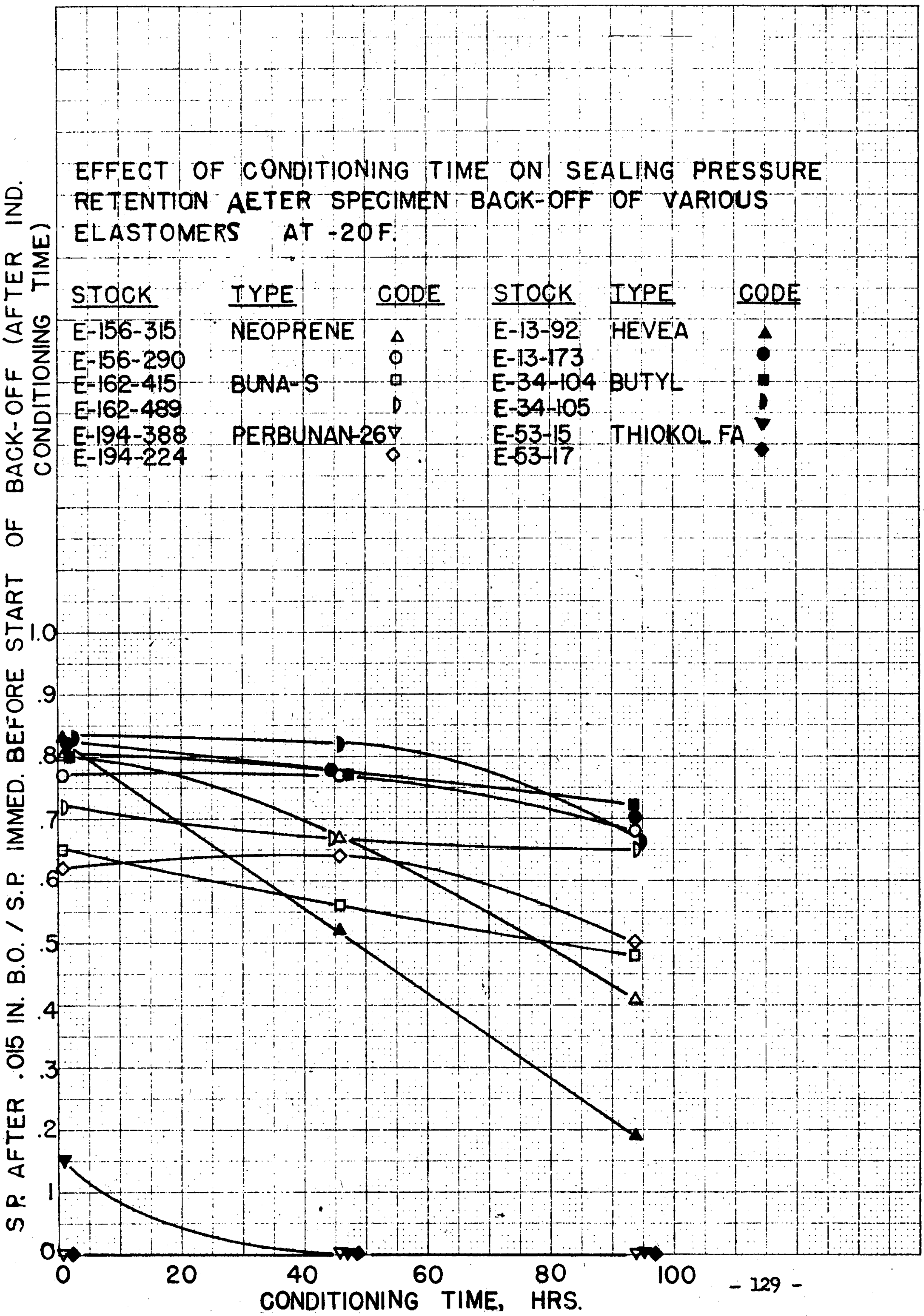

EFFECT OF CONDITIONING TEMPERATURE ON SEALING PRESSURE
RETENTION AFTER SPECIMEN BACK-OFF OF VARIOUS ELASTOMERS
STOCK TYPE CODE
E-156-315 NEOPRENE
E-156-290
E-162-415 BUNA-S
E-162-489
E-194-388 PERBUNAN-26
E-194-224
STOCK TYPE CODE
E-13-92 HEVEA
E-13-173
E-34-104 BUTYL
E-34-105
E-53-15 THIOKOL-FA
E-53-17
S.P. AFTER .015 IN. B.O./S.P. IMMED. BEFORE START OF BACK-OFF (AFTER 1HR. CONDITIONING)
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
0
-40
-20
0
20
40
60
80
CONDITIONING TEMPERATURE, F.

FIG. 25



EFFECT OF CONDITIONING TIME ON SEALING PRESSURE RETENTION AETER SPECIMEN BACK-OFF OF VARIOUS ELASTOMERS AT -20F.
STOCK TYPE CODE
E-156-315 NEOPRENE △
E-156-290 ○
E-162-415 BUNA-S □
E-162-489 ◗
E-194-388 PERBUNAN-26 ▽
E-194-224 ◇
STOCK TYPE CODE
E-13-92 HEVEA ▲
E-13-173 ●
E-34-104 BUTYL ■
E-34-105 ◗
E-53-15 THIOKOL FA ▼
E-53-17 ◆
S.P. AFTER .015 IN. B.O. / S.P. IMMED. BEFORE START OF BACK-OFF (AFTER IND. CONDITIONING TIME)
0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1.0
0
20
40
60
80
100
CONDITIONING TIME, HRS.

FIG. 26

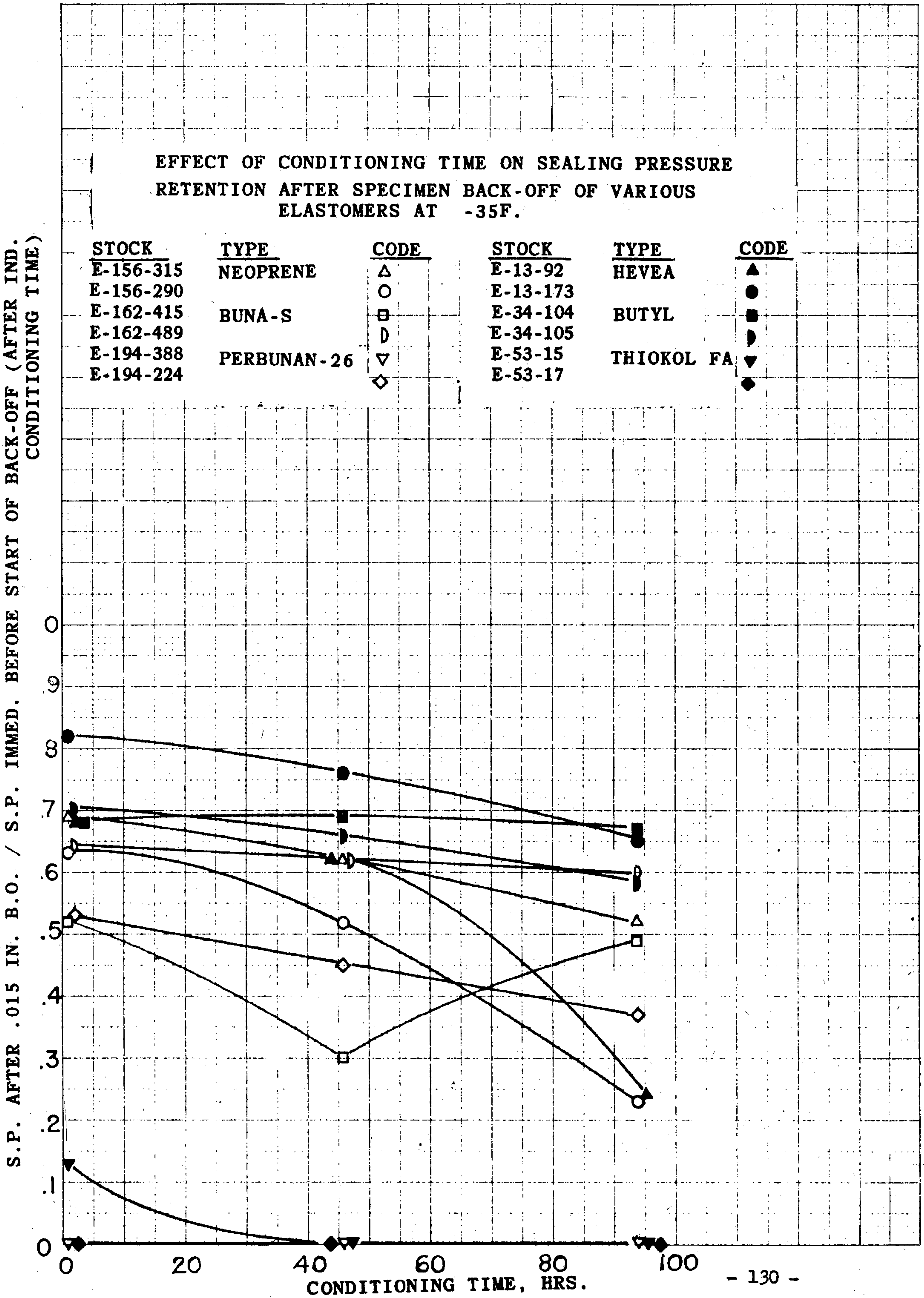

EFFECT OF CONDITIONING TIME ON SEALING PRESSURE
RETENTION AFTER SPECIMEN BACK-OFF OF VARIOUS
ELASTOMERS AT -35F.
STOCK
TYPE
CODE
E-156-315
NEOPRENE
E-156-290
E-162-415
BUNA-S
E-162-489
E-194-388
PERBUNAN-26
E-194-224
E-13-92
HEVEA
E-13-173
E-34-104
BUTYL
E-34-105
E-53-15
THIOKOL FA
E-53-17
S.P. AFTER .015 IN. B.O. / S.P. IMMED. BEFORE START OF BACK-OFF (AFTER IND. CONDITIONING TIME)
0
.9
8
.7
.6
.5
.4
.3
.2
.1
0
0
20
40
60
80
100
CONDITIONING TIME, HRS.

FIG. 27

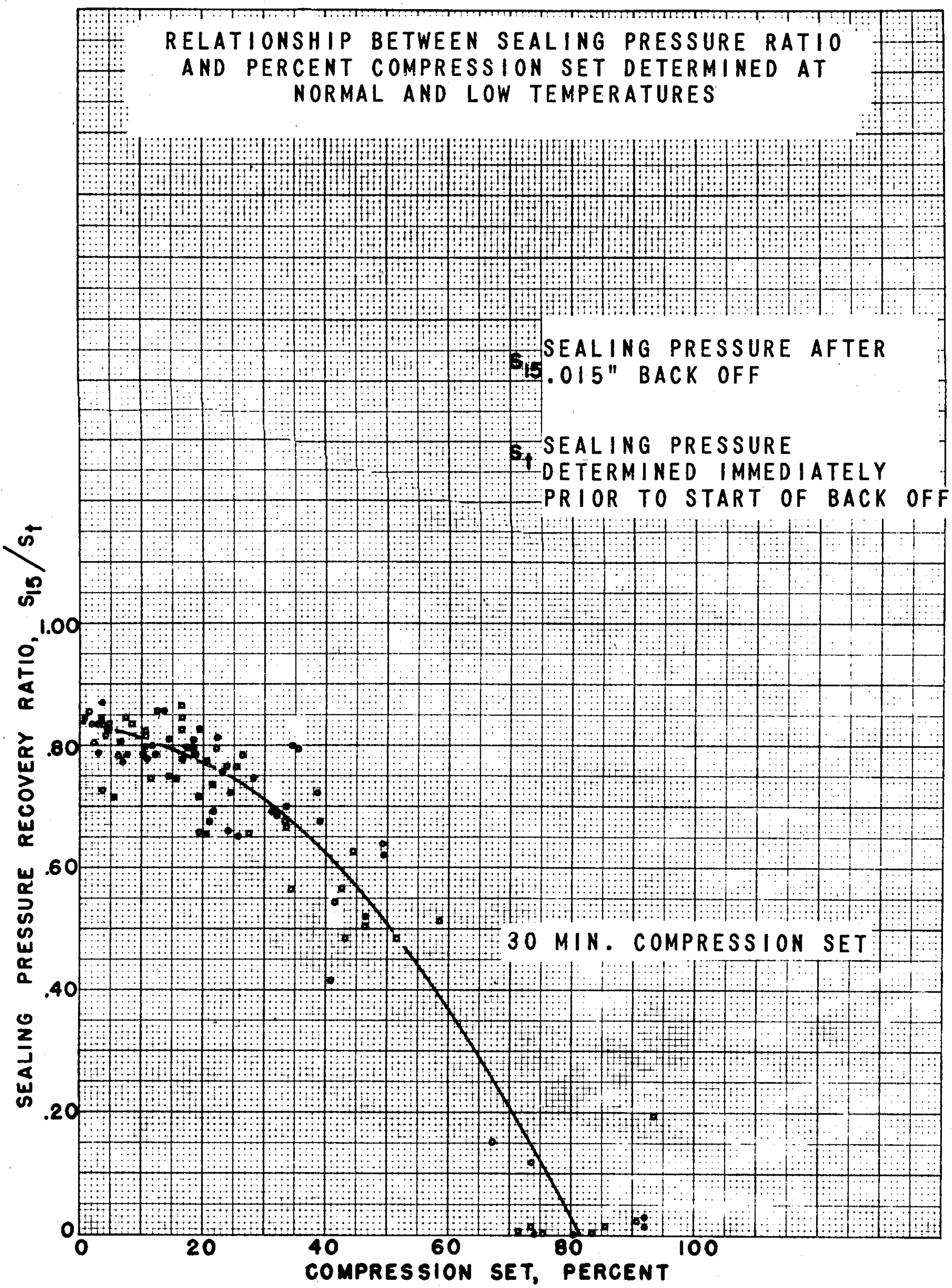
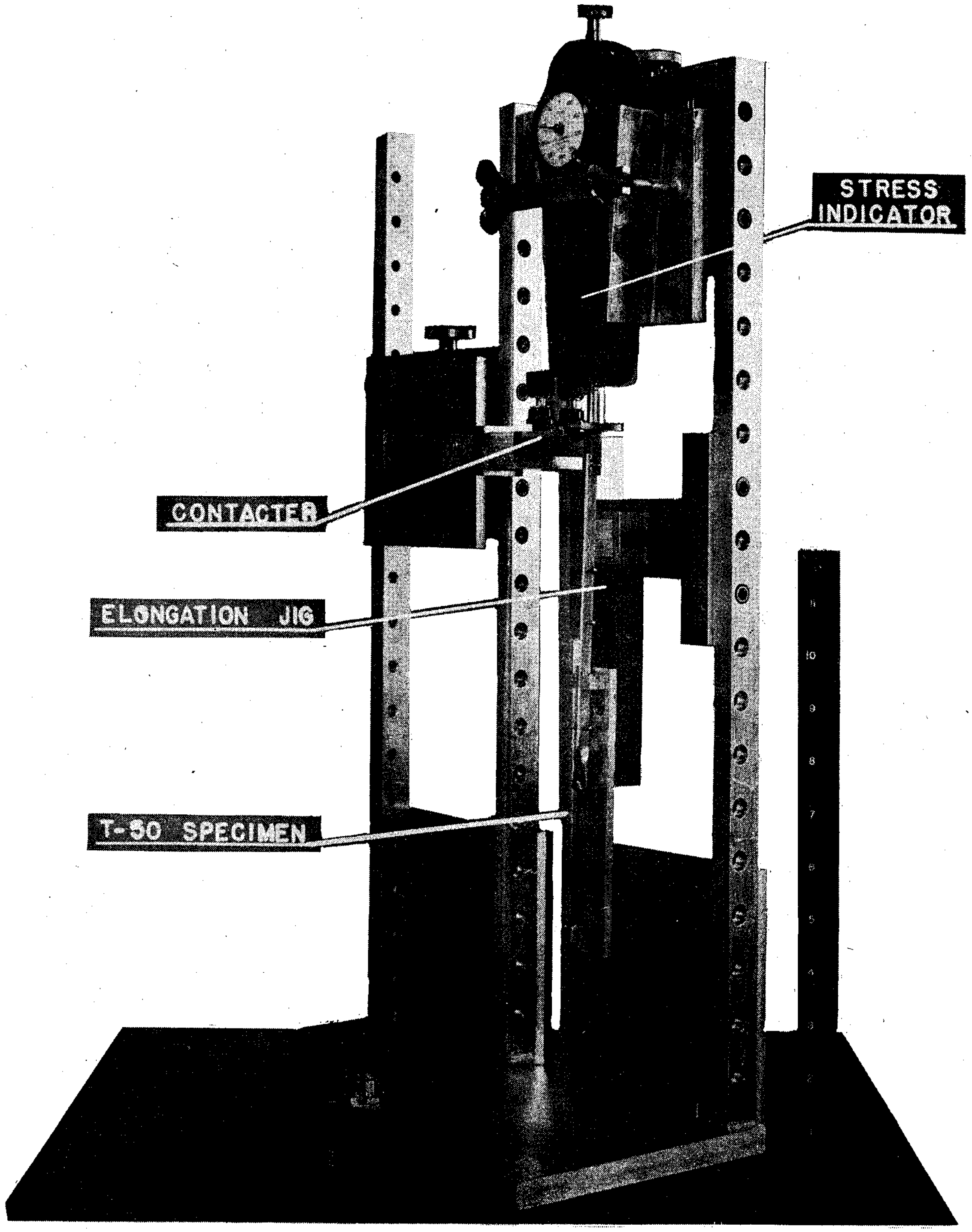

RELATIONSHIP BETWEEN SEALING PRESSURE RATIO AND PERCENT COMPRESSION SET DETERMINED AT NORMAL AND LOW TEMPERATURES
S_{15} SEALING PRESSURE AFTER .015" BACK OFF
S_t SEALING PRESSURE DETERMINED IMMEDIATELY PRIOR TO START OF BACK OFF
30 MIN. COMPRESSION SET
SEALING PRESSURE RECOVERY RATIO, S_{15}/S_t
1.00
.80
.60
.40
.20
0
0
20
40
60
80
100
COMPRESSION SET, PERCENT

FIGURE 28



DISCUSSION FOR ARMY ORDNANCE LOW TEMPERATURE RUBBER CONFERENCE

4 March 1952, Washington D. D.

BY

B. G. Labbe

University of Akron Government Laboratories

The low temperature properties of rubber and rubber-like materials have been investigated in many of the laboratories in this country. However, within the past few years, emphasis on this phase of testing has increased to the point where it engages the interest of practically all of the large laboratories. Although many of the proposed methods appeared to be of questionable value, the intense search for suitable tests, which could be utilized to predict low temperature serviceability, has justified most of the experiments.

By suitable tests, we refer to methods which would include the following points:

1. Ability to predict serviceability at low temperatures.
2. Good reproducibility.
3. Low-cost Equipment.
4. Ease of Operation.
5. Time required for test.

At the Government Laboratories we have investigated quite a few methods.

The first instrument utilized at our laboratories for determination of the low temperature properties of elastomer compounds was the Shore "A" durometer. Decadence between 5- and 30-second readings was plotted against test temperature and a definitely sharp break in the curve was assumed to be associated with the freeze point. Although the values ob-

tained show some correlation with those being obtained with the Gehman and Admiralty Hardness Testers today, the reproducibility was no better than plus or minus 5°F. Needless to say, we searched for an improvement of this method and subsequently, we tried the Tinius-Olsen hardness tester on specimens that were submerged in an acetone-dry ice bath. However, the data from this test were no better than those obtained with the Shore durometer.

Early in 1948, we believed that the most desirable low temperature test procedure would be one that was readily available to any laboratory at low cost, but which would still be efficient for prediction of low temperature performance of elastomeric products. Accordingly, we investigated a permanent set test, wherein a standard dumbbell strip was held at 200% elongation for 24 hours at minus 70°F. After the conditioning period, one end of the specimen strip was released and the percentage of retraction was noted at 5 seconds and at 30 minutes. The data obtained were not encouraging, and the experiment was discontinued.

With the advent of the Gehman flexibility tester, the Synthetic Rubber Division of the RFC requested us to investigate the possibility of installing a similar apparatus at the Government Laboratories. Through the cooperation of Dr. Gehman and his staff, an instrument which was identical with that constructed at the Goodyear Research Laboratories was built by our mechanical department. However, our first experience with this apparatus was very sad, in view of the fact that it required several months of training before non-technical employees could obtain fair reproducibility of

the test data. We also found that the 5-station apparatus could not meet our requirements with respect to work load. Consequently, a new 10-station tester was built. In this equipment, many improvements were embodied, such as the use of liquid nitrogen instead of dry ice as coolant, a temperature control device, and a removable torsion wire. Although this apparatus was much easier for the operator to handle, the reproducibility was not satisfactory. It was observed that the temperature varied as much as 15°F between the top and the bottom of the 1-inch test specimen. Accordingly, a small fan was built into the apparatus to equalize the temperature in the test chamber. Apparently this modification was the solution to our reproducibility problem and, in our opinion, would remove much of the variability encountered with the Gehman apparatus as it is marketed today.

Except for the freeze point, an empirical value, relative modulus values are dependent on the twist of the test specimen at 25°C. These results are sufficient for comparison of low temperature behavior of various polymer compounds, but for anyone who prefers absolute values, the actual stress in pounds per square inch required to twist the sample through one degree may be calculated.

As far as low temperature flexibility is concerned, we believe that the Gehman procedure is equal or superior to any other method in use at the present time. However, the ability to detect crystallization with certainty in all cases is questionable with the Gehman equipment. More will be said about this later.

In June, 1948, Sperberg, then with the Phillips Petroleum

Company, introduced the temperature retraction test, in which acetone was utilized as the coolant, to Subcommittee XXV of A.S.T.M. Within a few months, we conducted similar tests but used a "Sub-Zero" cabinet in which the air was cooled by dry ice instead of the acetone bath. Although correlation with Gehman results was indicated, the data were not encouraging. We realized that our temperature control was not sufficiently good, but nevertheless felt that we were getting more information from the Gehman apparatus with less effort than we could with the temperature-retraction test. On the other hand, developments achieved with the T-R test, whereby crystallization is indicated, have made this procedure more desirable than it first appeared to be. Recently, our mechanical department built a T-R apparatus which we believe embodies the desirable features of both the Phillips Petroleum and the United States Rubber Company equipment. One of the major drawbacks to application of this procedure for specification testing is that some of the mechanical goods compounds may not elongate to the 250% specified. However, this objection could probably be overcome by a slight revision of the specification.

In 1949, we determined the low temperature rebound characteristics of various polymer compounds measured by the Goodyear-Healy rebound and the Bashore resiliometer apparatus. The rebound test is not suitable for specification testing because it is slow and tedious and because the transition point which is generally, but not always, determined cannot be correlated with results of other tests. The Bashore resiliometer is superior to the Goodyear-Healy apparatus with

respect to ease of operation and defining transition points.

Of course, throughout the past six years we have conducted the compression set procedure, A.S.T.M. Method B. at subzero temperatures. Our setup for this test is not as efficient as some that we have seen, but in most installations the procedure involved seems to us to be a bit cumbersome. Nevertheless, we consider this method a necessity for specification testing of gasket materials. The equipment cost may be lower than for most of the low temperature testers.

The stress-relaxation of polymer compounds was studied and reported in 1948. The equipment required for these tests is relatively expensive and, therefore, is of value only as a research tool at present. Frankly, with our present knowledge of the test results, interpretation with respect to the efficiency of the various polymers is questionable.

We have conducted low temperature tests on inner tubes in a large cold room, but this work would probably be of little interest to this group.

From our point of view, the most important aspect of any low temperature apparatus is accurate and uniform temperature control, without which the best available apparatus would be of little consequence. The ability to obtain reproducible data and clear interpretation of the results are also essential.

About two years ago, the Synthetic Rubber Division sponsored a low temperature testing program which involved the Bureau of Ships Material Laboratory in Brooklyn, the Phillips Petroleum Company, and the Government Laboratories. Later, the United States Rubber Company laboratories in Passaic, New Jersey,

requested to participate in the crosscheck. The following nine polymers were selected for compounding:

1. Natural rubber.
2. GR-S polymerized at 122°F.
3. Emulsion polybutadiene made at 122°F.
4. Emulsion polybutadiene made at 14°F.
5. Sodium-catalyzed polybutadiene.
6. Sodium-catalyzed 75/25 butadiene/styrene.
7. Perbunan 26.
8. 85/15 Butadiene/styrene made at 122°F.
9. 80/8/12 Butadiene/isoprene/styrene made at 41°F.

Each of these polymers was compounded according to a standard test recipe and, in addition, GR-S, 85/15 butadiene/styrene, and Perbunan 26 were compounded in special gasket recipes.

The following testing methods were included:

1. Temperature retraction.
2. Low temperature extension.
3. Gehman low temperature flexibility.
4. Compression set.
5. Clash-Berg torsion test.
6. Material Laboratory British Admiralty Hardness test.
7. Stress-relaxation.
8. Shore "A" hardness.

We believe that you will be interested in the results obtained from this program. Dr. Helin will discuss the data obtained and interpretation of the results, as agreed upon by personnel from the four laboratories.

REPORT OF CROSS CHECK STUDY OF LOW TEMPERATURE EVALUATION METHODS FOR ARMY ORDNANCE LOW TEMPERATURE RUBBER CONFERENCE

March 4, 1952

BY

A. F. Helin

Government Laboratories

The test methods covered in our cross check study have been arbitrarily divided into two classifications; those which establish a freeze point and those which do not. In the first category are placed the Gehman, Clash-Berg, Low Temperature Extension, and Temperature Retraction or "T-R" tests. The second category includes the compression set, stress relaxation, rate of retraction, and hardness tests.

The Gehman and Clash-Berg tests determine over a range of temperatures the angular twist imparted to a specimen when torque is applied. In the T-R test method, an elongated specimen is conditioned at a low temperature, say minus 70°C, one end is released and allowed to retract as the batch is warmed up at a uniform rate. The Phillips method uses 50% elongation whereas the U.S. Rubber method used 250% elongation. The Phillips method requires plotting of the data and extrapolation of the curve to obtain a freeze point. The U.S. Rubber method determines the temperature at which definite percentage retraction values are obtained, namely, 10, 30, 50, and 70 percent, and evaluates the polymers from these data.

In the low temperature extension test, the specimen is conditioned at the test temperature, a weight is applied, and the percentage elongation of the specimen is measured after 30 seconds. After the test is repeated over a range

of temperatures, a curve is plotted and extrapolated to zero extension to establish a freeze point.

(Slide No. 1) The agreement among the freeze point values as determined by the Gehman, T-R and Low Temperature Extension tests is good, the deviation exceeding 5°C in only one case, 14°F polybutadiene, which will be discussed later. The agreement between Gehman data from two laboratories is excellent, values checking within 1°C in nine of the twelve cases with a maximum difference of 4°C. The freeze points obtained by Phillips T-R test agree well with the TR-10 values obtained by U.S. Rubber, the maximum deviation being 3°C. However, no freeze point could be established by the Phillips test for 14°F polybutadiene.

The behavior of 14°F polybutadiene in the various tests mentioned is considered to be the result of its strong tendency to crystallize under relatively mild conditions. This tendency causes an irregular Gehman curve and a T-R curve with a shape that cannot be satisfactorily extrapolated to obtain a freeze point. (Slides 2, 3, and 4) Thus, the freeze point of such a polymer is of doubtful significance. However, it should be recognized that in such cases the inadequacy of the evaluation due to the likelihood of crystallization is readily apparent and the necessity for evaluation by another method is thereby pointed out.

This should not be taken to mean that the Gehman test or the T-R test as conducted by Phillips is considered adequate for determining the crystallizing tendencies of a stock, for it is only with readily crystallizable stocks

that the irregularities in the curve are obtained. When such tendencies are suspected some confirmation can be obtained by repeating the test after conditioning for 22 or 94 hours at minus 35°F. Significant changes in the shapes of the curves for 14°F polybutadiene and natural rubber were noted, whereas the curves for non crystallizing compounds showed no such changes.

The temperature retraction data of the U.S. Rubber Company, when analyzed in the manner prescribed by them, indicate definite inferiority of the compounds made from natural rubber and 14°F polybutadiene. The wide spread in temperature between the TR-10 and TR-70 values, amounting to about 40°C as compared with 13° average spread for the other stocks, is taken as definite evidence of crystallization in the compound.

Freeze points were not determined on the basis of the Clash-Berg test, but this method so closely resembles the Gehman method in principle that it will be discussed at this point. Direct comparisons between the Gehman and Clash-Berg test methods were carried out by the Material Laboratory, and the flexural moduli of the specimens were calculated. In most cases discrepancies amounting to more than 200% were noted between the values obtained by the two methods. The major portion of the discrepancies was attributed to the effect of friction in the pulleys and bearings of the Clash-Berg apparatus which was entirely enclosed in the cold chamber during the test.

In general, the data from the various freeze point methods gave the polymers the same relative ratings. Of course, the lowest temperature at which a stock can be put into prac-

tical service will be well above its freeze point, since the modulus at temperatures approaching the freeze point is unduly high. The significance of the freeze point lies in the fact that polymers or compounds can be evaluated on a basis of their inherent potentialities rather than their performance at a specific temperature. The freeze point is hence more fundamental and for routine screening of polymers is more desirable than other types of evaluation. However, it is unquestionably true that in the long run, a test designed to approximate the service conditions of the end product should be the most valuable type in setting up specifications.

The tests which do not establish a freeze point were carried out by the following procedures:

The compression set test procedure is well known and need not be described.

The stress relaxation equipment measures, on a Foxboro Dynalog, the actual pressure that a compressed test specimen exerts on a strain gage. For the test as conducted in the present investigation, the test specimen at minus 35°F is placed on a lower anvil which is raised to an upper anvil by means of a crank and gear arrangement compressing the test specimen by 40%. After recording the stress decay during a period of 2, 22, or 94 hours, the anvils are opened 2% of the original test sample height. This operation causes a sharp drop in pressure which gradually builds up again as the sample recovers. The extent of the pressure drop and the speed and amount of recovery are considered to be the most significant observations provided by the test.

The Material Laboratory Admiralty hardness tester, which applies a dead weight load on an indentor stem having a hemispherical tip, is similar to the Pusey and Jones and the Tinius Olsen hardness testers. The depth of indentation of course is inversely related to the hardness. The standard Shore A Durometer was utilized for testing samples first at 80°F and again after conditioning for 22 hours at minus 35°F.

In the rate of retraction test a specimen is elongated 50% of its original length and is conditioned for the desired time at the test temperature. One end of the specimen is released and the retraction at definite time intervals is noted.

(Slide No. 5) Direct comparisons between compression set tests conducted by the Government Laboratories, the Material Laboratory and the U.S. Rubber Co. on samples conditioned 94 hours at minus 35°F under 40% compression gave an average deviation of 4.2% for 10 second set values and 4.4% for 30 minute set values. Maximum differences between laboratories were 10.8% for 10 second set values and 25.8% for 30 minute set values. From these results it might be concluded that the reproducibility of the test method is poor, but since past experience has indicated much better reproducibility, the variations were probably caused by slight differences in techniques. The importance of a standardized technique for the test is indicated from comparison of the data obtained by the Materials Laboratory, the Government Laboratory, and the U.S. Rubber Co., with those of Phillips who used different deflections and conditioning periods. Although many of the results were similar, large variations

were observed in a number of cases. If the compression set values obtained by the Materials Laboratory, the Government Laboratory, and the U.S. Rubber Co. are averaged, a good correlation with the TR-70 values is noted, the deviation from straight line relationship not exceeding 3°C except for natural rubber and Perbunan whose compression set values showed wide difference between laboratories.

The stress-relaxation test which was conducted only by the Government Laboratory is similar to the compression set test in principle but provides more complete data since the load is automatically recorded throughout the test period. The relative ratings of the polymers were much the same as those obtained in the other low temperature tests. However, since its correlation with polymer serviceability has not been established, the usefulness of the recovery data is greatly restricted. Furthermore, inconsistencies and lack of reproducibility have indicated that both the procedure and the equipment should be improved.

The data of the Material Laboratory Admiralty hardness tests are best interpreted by comparison with a limiting value for hardness indentation below which the material is judged to be unsuitable for a specific purpose. For example, for Navy door and hatch gasket stock, the minimum hardness indentation for service at minus 35°F is 1.00 mn. Some s tocks which by other tests showed good low temperature properties would have been rated unserviceable for this purpose by the Admiralty test, since their intrinsic hardness is high, approaching, at room temperature, the limit specified for minus 35°F.

The Material Laboratory carried out a mathematical analysis of the relationship between the Gehman values and the hardness indentation values obtained with their equipment and arrived at the simple expression

$$E = \frac{820}{H^{1.36}}$$

where E is the flex modulus in lb/sq in. and H is the indentation in mm.

The Shore A durometer hardness values provided little information of consequence. A direct comparison of the durometer values of two or more polymers at minus 35°F has little significance in view of the fact that they may have widely different durometer values at room temperature. If judged on the basis that a high percentage increase in hardness in going from room temperature to minus 35°F is detrimental to efficiency at low temperatures, a rating might be obtained, but because of the meager data available the reproducibility of such rating is not known.

Our opinions regarding the merits of the individual tests as a result of this study are as follows:

GEHMAN FLEXIBILITY

The Gehman procedure is capable of good reproducibility both within a laboratory and between laboratories. Although the test procedures were the same, the actual instruments used at Phillips and at the Government Laboratories differed in capacity, method of temperature control and type of coolant. The values reported by the Gehman test do not show crystallization, although the shape of the Gehman curve may indicate this property.

CLASH-BERG

Because the tests conducted with this apparatus were performed in a manner differing somewhat from the accepted procedure, inasmuch as the working parts of the apparatus as well as the test specimens were subjected to the low temperatures, a fair evaluation of this test method cannot be made. On the basis of the performance reported by the Material Laboratory, working parts of this apparatus suffered too much friction for satisfactory reproducibility and proper evaluation of the polymers. However, the procedure reported by the United States Rubber Company is claimed by them to be highly reproducible.

TEMPERATURE-RETRACTION

The T-R method is advantageous, inasmuch as specimens are conditioned under stress, thereby providing a means of determining crystallization. However, the crystallization, as determined by the Phillips' method, is not defined numerically but is deduced from a graph of the data. The method of the United States Rubber Company provides a more definite evaluation of crystallization, but the elastic limit of a compound may affect some results. As carried out by standard test procedures, the acetone bath coolant does not produce the low temperatures desirable for some of the stocks. However, the methanol-dry ice coolant utilized by U.S. Rubber is satisfactory for temperatures as low as minus 75°C.

EXTENSION TEST

The application of a load to elongate a conditioned test specimen supplies data similar to those obtained with the T-R and Gehman instruments. Plotted curves tend to show

crystallization but the tendency is not well defined. The procedure for the extension test is somewhat cumbersome but a group of 20 samples can be tested in a day by one operator.

RATE OF RETRACTION

The rate-of-retraction test is fairly reproducible and can differentiate between the phenomena of cold hardening and crystallization. The test should be run at several temperatures for complete polymer evaluation. The results correlate with those of the compression-set test. As conducted, the test procedure is relatively simple.

COMPRESSION SET

This is probably one of the most widely used low temperature test methods. The reproducibility between laboratories is usually better than that found in the present investigation. The recovery factor at various time intervals, after deflection and release, is of vital importance in the selection of many gaskets and automotive parts. The test apparatus is less convenient to handle than other tests and the duration of the test is too long for a quick evaluation.

ADMIRALTY HARDNESS

The Material Laboratory modification of the Admiralty tester appears more efficient than any previous method involving indentation measurements. From the available data the reproducibility of results is good. The test procedure is time-consuming only as to the time required for conditioning samples. The inaccuracies usually ascribed to dead load hardness testers due to friction in the dial gage assembly is eliminated by means of a small vibratory motor.

SHORE A DUROMETER

This method is useful only because of the availibity of instruments. Previous investigations with the Durometer at the Government Laboratories on the hardness variations with temperature resulted in very erractic data. On the basis of the results obtained in this program, no evaluation of reproducibility can be made.

STRESS-RELAXATION

The stress-relaxation test provides more information than compression set and produces data showing recovery values based on the applied load and the retention of the initially applied stress during any part of the conditioning period. The apparatus is expensive, and the importance of relaxation to various service articles is not fully known. With the present equipment, reproducibility is not as good as it should be because of minor faults in the construction of the jigs and the method of manually applying the load.

The final conclusions from this survey may be summed up as follows.

At the present time the T-R, Gehman, and compression-set tests appear most useful for a general evaluation of a polymer. For selecting polymers for special purposes, test procedures approximating the service application of the polymer are desirable.

FREEZE POINT AND RETRACTION VALUES, MINUS °C

Polymer		Government Laboratories Gehman F. P.	Gehman F.P.	T-R F.P.	Extension F.P.	U.S.Rubber TR10	TR70
1	Natural Rubber	58	59	56	61	56	15
2	GR-S	49	50	47	51	48	37
3	122°F PolyBD	73	76	72	72	71	53
4	14°F	67	70	--	71	56	15
5	Na PolyBD	42	43	40	43	42	32
6	Na 75/25 BD/S	26	26	24	25	26	15
7	Perbunan 26	31	30	28	31	29	20
8	85/15 BD/S	61	62	59	62	61	49
9	80/8/12 BD/I/S	59	60	56	60	59	48
10	GR-S[a]	64	64	61	63	63	47
11	85/15 BD/S[a]	71	71	69	70	70	54
12	Perbunan 26[a]	52	48	48	51	49	36

[a] Mixed in a special gasket recipe.

Slide #2

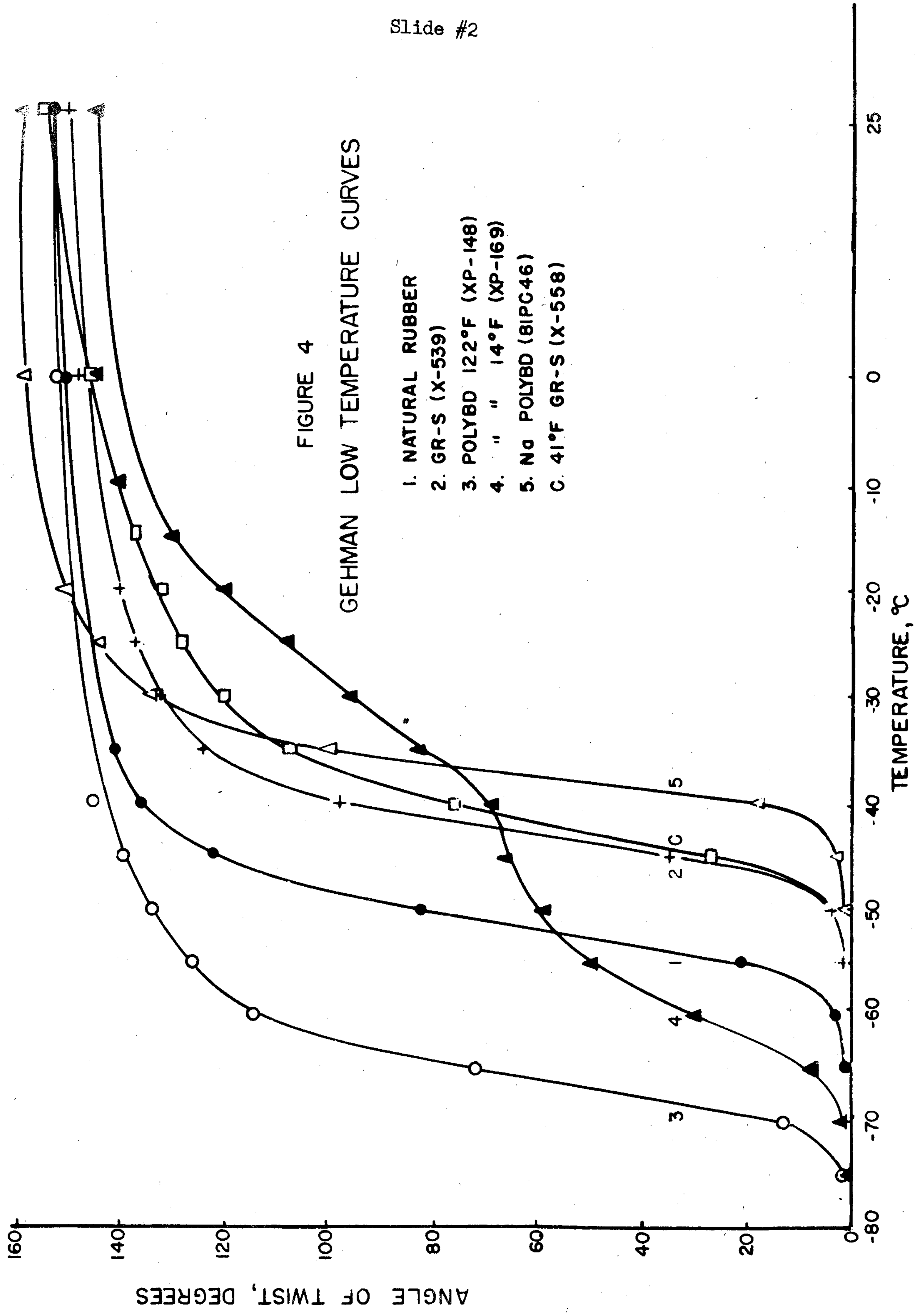
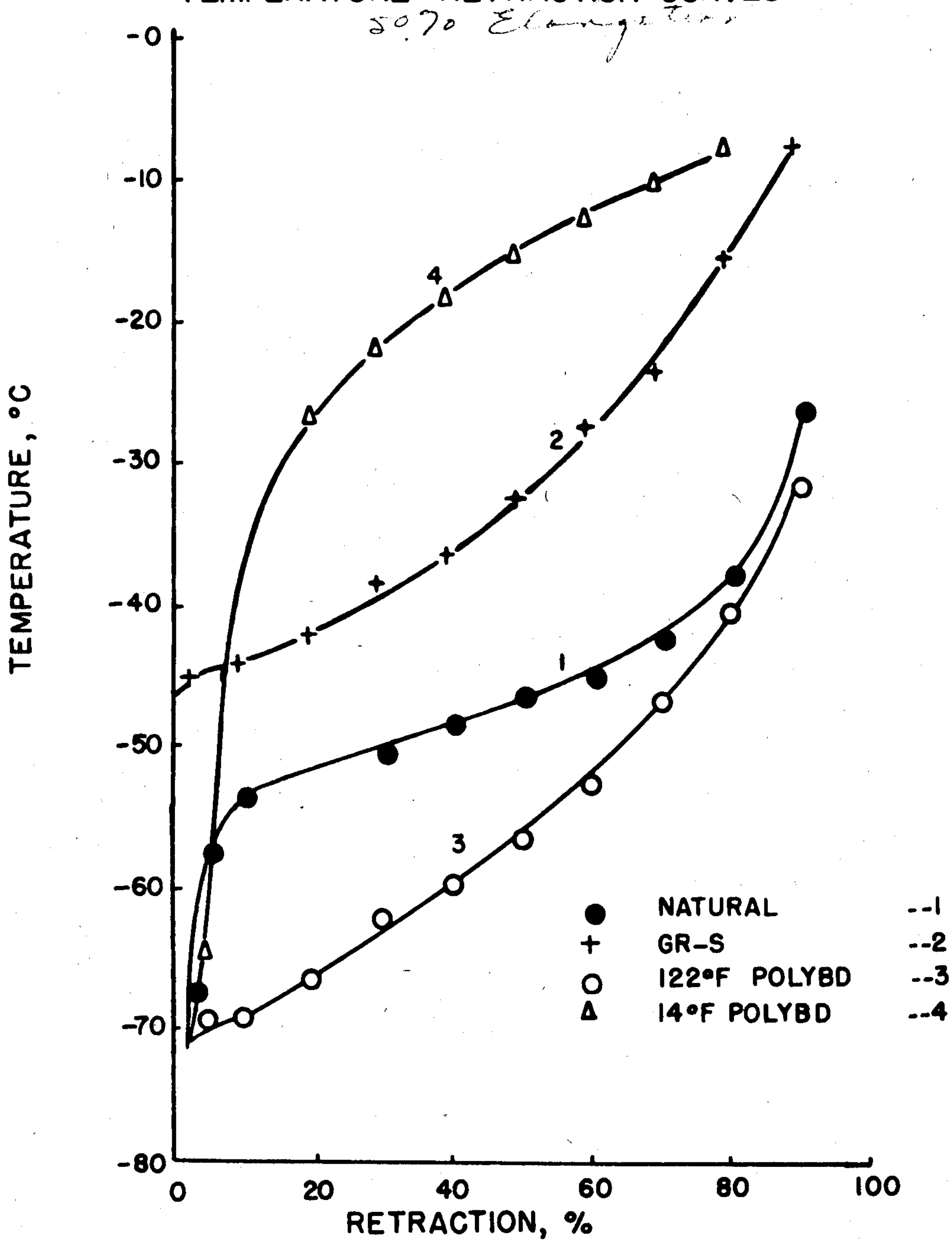


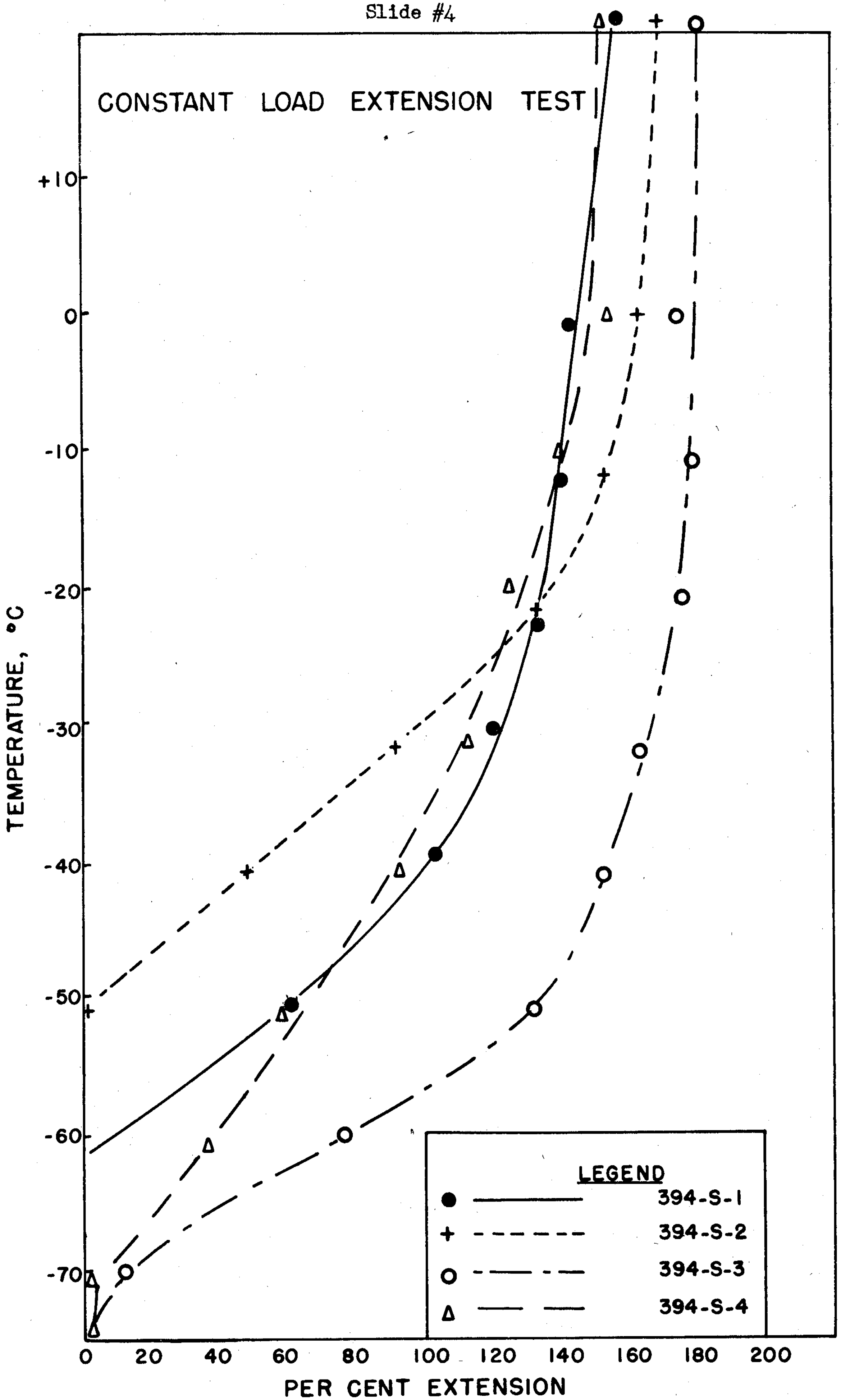
FIGURE 4
GEHMAN LOW TEMPERATURE CURVES
1. NATURAL RUBBER
2. GR-S (X-539)
3. POLYBD 122°F (XP-148)
4. " " 14°F (XP-169)
5. Na POLYBD (81PC46)
C. 41°F GR-S (X-558)
TEMPERATURE, °C
ANGLE OF TWIST, DEGREES
-80
-70
-60
-50
-40
-30
-20
-10
0
25
0
20
40
60
80
100
120
140
160
1
2
3
4
5
C

Slide #3

FIGURE I

TEMPERATURE-RETRACTION CURVES




CONSTANT LOAD EXTENSION TEST
TEMPERATURE, °C
+10
0
-10
-20
-30
-40
-50
-60
-70
0
20
40
60
80
100
120
140
160
180
200
PER CENT EXTENSION
LEGEND
394-S-1
394-S-2
394-S-3
394-S-4

COMPRESSION - SET DATA

SPECIMENS CONDITIONED 94 HRS. AT MINUS 35°F

	Polymer	Material Laboratory	Government Laboratory	U. S. Rubber Co.
		30-Minute Set Values, %		
1	Natural Rubber	47.5	40.4	66.2
2	GR-S	46.4	53.4	49.1
3	122°F PolyBD	21.9	30.3	22.6
4	14°F "	94.2	98.1	94.9
5	Na "	52.0	64.6	64.1
6	Na 75/25 BD/S	96.1	98.0	97.8
7	Perbunan 26	90.0	98.3	96.8
8	85/15 BD/S	20.3	26.1	24.6
9	80/8/12 BD/I/S	21.2	28.0	20.0
10	GR-S[a]	24.9	33.1	27.3
11	85/15 BD/S[a]	18.3	26.0	19.6
12	Perbunan 26[a]	29.3	36.5	52.5

[a] Special gasket stock recipe.

REVIEW OF THE STATUS OF SAE-ASTM SUBSECTION IV-L FINDINGS ON LOW TEMPERATURE TESTS

BY

Dr. Hanson

Rock Island Arsenal

PROPOSED TENTATIVE RECOMMENDED PRACTICE FOR DETERMINING LOW TEMPERATURE PROPERTIES OF AUTOMOTIVE RUBBER COMPOUNDS

Scope

1. This recommended practice covers types of tests recommended for properties of automotive rubber when exposed to low temperatures.

Types of Test

2. This recommended practice covers four types of tests for the properties most often required, as follows:

Flexibility

Compression Set

Brittleness

Hardness

Note: Each method is to be used to indicate the one property tested. It should be emphasized that the property being measured should be the one that is critical in the application of the rubber compound.

Conditioning

3. Prior to testing, the rubber shall be conditioned in accordance with the ASTM Recommended Practice for Conditioning of Rubber and Plastic Materials for Low-Temperature Testing (ASTM Designation D832).

Methods of Test

4.a. Flexibility - The flexibility of an elastomer is its pliancy or the ease with which it is bent. Stiffness is

usually thought of as lack of pliancy. As the ease or difficulty with which an elastomer can be bent is indicated by the stress required to produce a given strain either in flexure, torsion or extension, the modulus of elasticity or Young's modulus can be directly interpreted in terms of Flexibility. As modulus is the ratio of stress to strain, it may be calculated from the results of a number of test methods. If tests are to be correlated between laboratories using different methods, the results should be calculated to modulus of elasticity. Following are suggested methods:

(1) The Standard Method of Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (ASTM Designation D797).

(2) Method of Measuring Low Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus (ASTM Designation D1053).

(3) Stiffness Properties of Non-rigid Plastics as a Function of Temperature by Means of a Torsional Test (ASTM Designation D1043).

b. Compression Set - The recovery after compression at low temperature evaluates the suitability of a material for gaskets and packings since the sealing efficiency is dependent on the force exerted between the confining surfaces due to the elastic recovery properties in the rubber. The proposed "Tentative Method of Test for Low Temperature Compression Set of Vulcanized Elastomers", is recommended. (See Note)

c. <u>Brittleness</u> - The limit of serviceability of a rubber compound is indicated by its brittle temperature. However, it is usually sufficient to know that a rubber compound is not brittle above a given temperature. Therefore, an impact test is made at a specified temperature and if failure does not occur, the rubber is considered satisfactory. For this test, the Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (ASTM Designation D746-44T) modified to test at specified temperatures and conditioning time in air, is recommended.

d. <u>Hardness</u> - This test is a measure of resistance to indentation. It is sometimes used as an indication of other properties but such relationship is indefinite. For measuring hardness, a durometer, calibrated and used in accordance with the Tentative Method of Test for Indentation of Rubber by Means of a Durometer (ASTM Designation D676), is recommended. However, the specified durometer will not differentiate between stocks with hardness readings of 95 or over. Therefore, hardnesses in this range shall be recorded as, "greater than 95".

NOTE - The apparatus and procedure specified in ASTM D395, Method B, will be used with the following exceptions:

<u>Apparatus</u>

Cold Box - A dry ice unit or a mechanical cold box preferably of the "top opening" type and capable of temperature control of $\pm 2^{\circ}$F., thus conforming to D-832. The test chamber shall be equipped with a vise for holding the compression set jig.

Procedure

Only one specimen shall be used per jig and it shall be placed in the center of the plate.

Within 30 minutes after the jigs are loaded, they shall be placed in the low temperature cabinet, the temperature of the cabinet to be -40°F. or -65°F.

The conditioning period shall be either 22 or 94 hours.

Approximately one hour before the conditioning period is over, the dial gage (lubricated with Silicone lubricant) shall be placed in the test chamber and one of the set jigs clamped in the vise or a quick opening clamp provided in the low temperature chamber. Suitable gloves shall be used for all operations in the test chamber. At the end of the conditioning period, the nuts shall be removed from the jig after which the vise is released and the stop watch started simultaneously. Thickness of pellets shall be measured 10 seconds and 30 minutes after release from vise and recorded. Since the test is conducted at a specific temperature ±2°F., the schedule of opening the jigs shall be such that the test chamber will stay within this temperature tolerance.

Check Test - Tests shall be run in duplicate and the results should agree within 5.0 percent.

Report

The report shall include the following:

1. The original thickness, t_o,
2. The percentage compression of the specimen actually tested,
3. The thickness of the test specimen 10 seconds, $t_{1_{10}}$, and 30 minutes, $t_{1_{30}}$, after removal from

the clamp,

4. The compression set expressed as a percentage of the original deflection, shall be calculated as follows:

$$c = \frac{t_o - t_1}{t_o - t_s} \times 100$$

Where:

c = Compression set expressed as a percentage of the original deflection, and

t_s = Thickness of the spacer bar used.

5. Temperature used,

6. Conditioning period.

REVIEW OF WORK OF ASTM TASK GROUP B OF COMMITTEE EI

BY

M. Boor

Quartermaster Corps

At the June 1949 meeting, the Navy requested ASTM to sponsor a survey of the elastomer and plastics industries directed toward:

Standardization of test methods

Correlations between methods measuring similar properties

Publication of new test methods or schemes of evaluation

All of the above were in special relation to measurement of pertinent properties at low temperatures.

This project was assigned to Task Group B of Committee El, under the Chairmanship of Mr. R.S. Havenhill of the St. Joseph Lead Company who appointed the writer Chairman of a Sub-group to organize this questionaire and summarize the replies as a guide to further action.

Four meetings were held in 1949 at which the draft of the questionnaire was agreed upon. It included 14 different principal categories of rubber testing, subdivided into a total of 98 different, specific methods. This questionnaire was sent (April & May 1950) to approximately 160 firms, laboratories, and institutions concerned with rubber testing. It also included specific questions on frequency of use, modifications, and invited submission of any observed correlations among various tests of a single property, or among different properties.

After the usual follow-up letters to the delinquent

laboratories, and 7 meetings during 1950, the task of summarizing and graphing the returns from the questionnaire was completed during the fall and winter of 1950 and presented to the Task Group at the Cincinnati meeting on 8 March 1951.

Fifty-six (56) replies were received to the questionnaires sent out (approximately 160); nineteen (19) reported no tests at other than room temperature. The summary of replies from the remaining 37 were tabulated in a chart which was distributed to the members of the committee. The 37 replies were from the following:

Rubber manufacturers	11
Material suppliers	9
Government laboratories	8
Plastic manufacturers	5
Wire and insulation manufacturers	3
Consumers	1

The conclusions, quoted from Mr. Scoville's report of 6 March 1951:

1. "...In general the type of tests used by the various contributors is dependent on the nature of their end products, with the government laboratories showing extensive interest in the entire range.

2. The most interest in methods was shown in the following order:

   A. Stiffness

   B. Brittleness

   C. Hardness

   D. Stress Relaxation

E. Swelling and Shrinkage

F. Tensile and Elongation

G. Creep

H. (Permeability
(Resilience

I. Tear

J. Shock

K. Fatigue

L. Surface

M. Wear

3. There was no unanimity of tests for measuring closely related properties, distribution being spread widely between known methods.

4. In general A.S.T.M. methods are widely used.

5. Very little was shown on the returns that any correlation might exist between the various methods most widely used. However the order in which the various properties are investigated might indicate that there is a definite desire to show a basic characteristic of the elastomers at low temperatures. Since the various methods to show certain properties are so widely varied it is doubtful if any conversion factors would be obtainable, as many methods are not a measure of a single property..."

The group met again 17 May 1951 under the new Subgroup Chairman, Mr. F.M. Gavan of Armstrong Cork Co., and agreed on definitions (for the purpose of the committee) of the properties most universally measured, namely, stiffness.

It was found that there are nine (9) methods now in use to measure various indices of the effect of applied force to elastomers. These are submitted on p.164.

It was agreed that three representative materials, gum, GR-S, and polyethylene, would be prepared by one source and submitted to those laboratories which signified their agreement to test the three materials by the method in which they were most expert, at the following temperatures, 73, 20, -20, -40, -60, -80°F.

Each laboratory contributed the data on the precise manner of performing the test, time intervals involved, and method of calculating results. These data are tabulated in a summary prepared by Mr. Scoville, dated 29 Nov 1951.

The list of collaborators and the methods to be used by each are attached, (p.164).

The samples, prepared by Mr. B.G. Labbe of the Government Labs were distributed during December 1951.

Some returns have been received and a progress report is to be presented at the Cleveland meeting of the Task Group during the week of 3 March. A partial report of the work at the Philadelphia QM Research & Development laboratories is appended.

Acknowledgment is made to members of this Task Group and especially to Messrs. Havenhill, Chairman of the main Task Group, Labbe of the Akron Government Labs who prepared and distributed the samples, and to Mr. Scoville of U.S. Rubber who had the formidable task of summarizing and tabulating the results of the questionnaire, corresponding with collaborators, tabulating the basic principles of each test and, we hope will continue his good work of collecting and summarizing the results of the cooperative tests on stiffness.

Low Temperature Testing of Elastomers and Plastics

Stiffness Methods	Test Laboratories
A. Tensile Modulus ASTM - D-412	
A-1 Scott	1. Government Laboratories - B. G. Labbe
A-2 Instron	2. Goodrich Research - R. Shearer
A-3 Tate-Emery	3. Philadelphia QM Depot - L. Boor
	4. DuPont Company, Poly-chemical-A.C. Webber
	5. Bakelite Company - W.A. Zincow
B. Olsen ASTM-D-747	Philadelphia QM Depot - L. Boor
	6. Materials Lab-N.Y. Naval Shipyard-C.K. Chatten
	Bakelite Company - W.A. Zincow
C. Firestone-Liska ASTM-D-797	7. Firestone Tire & Rubber Co. - F.S. Conant
	Materials Lab-N.Y. Naval Shipyard-C.K.Chatten*
	8. Naval Air Exp.Station-C. E. Granger -- Aeronautical Materials Labs
D. Compression ASTM-D-575	9. Manhattan Rubber Div. - S. Doner
	10. U.S. Rubber Co. - W. E. Scoville, Jr.
	Materials Lab-N.Y. Naval Shipyard - C.K. Chatten
E. Cantilever Beam ZZ-R-601	Manhattan Rubber Div. - S. Doner
	11. Mare Island Navy Yard - R. E. Morris
	U. S. Rubber Co. - W. E. Scoville, Jr.
F. Gehman ASTM-D-1053-49T	12. Goodyear Research - Dr. Gehman
	Government Laboratories - B. G. Labbe
	Mare Island Navy Yard - R. E. Morris
G. Clash Berg ASTM-1043-49T	Bakelite Company - W.A. Zinzow
	13. Rock Island Arsenal - A.C. Hanson
	Mare Island Navy Yard - R. E. Morris

Stiffness Methods	Test Laboratories
H. Taber Stiffness	Rock Island Arsenal - A. C. Hanson
	14. U.S. Army Ordnance - G. Reinsmith
J. Tensile Modulus	Goodrich Research - A. E. Juve

*Special Equipment

TO: ASTM Committee E-1, Task Group B

Low Temperature Testing of Elastomers - Plastics

As part of a Round Robin to examine various methods of measuring the stress-strain relationship in elastomers and plastics at various temperatures, the Chemicals & Plastics Division of the Philadelphia QM Research and Development Laboratories submits herewith the data on a portion of its assigned task, including:

a. Olsen Stiffness tests on the three selected polymers at six (6) temperatures.

b. Tensile stress-strain curves up to 100 percent elongation made on the Instron tester at five (5) rates of extension at one temperature, 73°F. The corresponding tensile curves at the other five temperatures will be forthcoming on completion of the testing machine enclosure.

Olsen Stiffness Tests

One of the tools for evaluating the effect of force on a material is the Olsen Stiffness Tester in which a sample in the form of a cantilever is slowly bent against a pendulum lever dynamometer; the data consist of readings of moment against angle. Its use for polymeric materials is described in ASTM D 747-485. The stiffness in flexure is derived from a formula which includes span, width, thickness, the moment and the angle. The time sensitiveness on non-elastic materials is partially recognized by the stipulation that the rate of rotation of the beam shall be 60 degrees per minute. However, the angle at which the stiffness is calculated is not defined, but a tangent is drawn

to the initial straight line portion of the curve. Those who have worked with this method will immediately recognize the difficulties in locating a tangent to a line which may have a convex or concave initial portion due to non-linear behavior at small increments of bending or to artifacts such as surface roughness and curl or twist in the sample. To overcome this objection, a method has been proposed which utilizes an increment of load between two fixed angles such as 5^{o} and 15^{o}, similar in principle to the Rockwell Hardness Value.

Two exhaustive studies of this instrument for this purpose served as background for this work, namely:

Report 4855-7, Material Laboratory, New York Naval Shipyard.

Another investigation was presented by Stechert, in the ASTM Bulletin No. 157, March 1949. He showed on a single rubber sample that the values of E (modulus) obtained were dependent on:

1. Specimen thickness
2. Span Length
3. Angle
4. Pendulum Weight
5. Width

Since the calculated formula for E is based on perfectly elastic behavior, any departure from perfect elasticity invalidates this relationship. His conclusions were:

a. "The greater the span, the larger is E"

If the same angle of bend is assumed, this is reason-

able since the stress on the outside fibers of a long span is less than with a short span, and most polymeric materials decrease in modulus with increase of strain.

b. "The greater the specimen thickness, the small the E" Again, if the same angle is assumed, this is logical since the stress increases as a power function of the thickness.

c. "There was little variation in E with specimen width"

d. "E was approximately constant with different pendulum weights"

The conclusions c and d are proper from the data presented; however, they do not take into account the angle, which is a measure of strain. It is obvious that if weight A at a scale reading of 100 gives an angle of 60°, then a weight $\frac{A}{4}$ will give only about 15° of bend at the same load scale reading. If the material tested is perfectly elastic, the E will be the same in both instances; however, if E decreases with strain, the test with the angular bend of 15° will give a higher E than the one with 60° strain. Recognizing these sources of variation of the "E" obtained by this method, the following approach was followed:

Considering the wide range of moduli to be measured, from pure gum at r oom temperature to polyethylene at -80 (from 200 to 150,000 Psi, a range of 750 to 1), and the desirability of covering the whole range on one instrument, preliminary tests were made on the $\frac{1}{2}$ inch-pound and 6 inch-pound machines. It became immediately obvious that to use the low capacity machine would necessi-

tate changing span which would bring in an undesirable variable.

It was found that with the 6 inch-pound machine, the span could be kept constant; any specimen could be bent to a nearly constant angle by varying the mass and, when the upper limit of mass was reached, the width could be decreased to obtain a curve in the desired range.

The requirement that a sample of a fixed span be bent to approximately the same angle during a test assures some control of two important variables:

a. The degree of strain in the sample.

b. Since the head rotates at 60°/min., this implies that the rate at which the sample is bent is nearly constant.

Practically, this requires that the curve drawn from the data, when plotted on the usual graph of percent load scale vs angle, shall be a nearly 45° line. This is possible only for elastic or nearly elastic materials. For materials with yield points where the curve changes shape rapidly, the initial portion should approximate a 45° angle.

All specimens were 2" long, and the span was held constant at ½". The weights and widths used for each material at each temperature were as follows:

Table I

TEMPERATURE °F	GUM Width	GUM Mass	GR-S Width	GR-S Mass	POLYETHYLENE Width	POLYETHYLENE Mass
73	1"	.1	1"	.35	0.250"	3.1
20	1"	.1	1"	.50	0.250"	6.0
-20	1"	.1	1"	.50	0.250"	6.0
-40	1"	.1	1"	.85	0.250"	6.0
-60	1"	.1	1"	2.00	0.250"	6.0
-80	1"	.6	0.5"	6.00	0.075"	4.6

Testing

Three separate specimens of each material were tested at room temperature. After overnight recovery, the same three samples were then conditioned at the indicated test temperature for 1 - $1\frac{1}{2}$ hours and tested.

They were then allowed to recover at $73^{\circ}F.$ overnight, and re-conditioned 1 - $1\frac{1}{2}$ hours at the successively lower temperature. Tests at $73^{\circ}F.$ were made in a conditioned room; all others were made in a dry ice box with circulation. The temperatures were accurate to $\pm$ $2^{\circ}F.$

After testing a sample at low temperature and allowing it to recover at $73^{\circ}F.$ at least overnight, no permanent deformation due to previous testing was observed.* The data are shown in the attached graphs. Stiffnesses were calculated from the relationship.

$$E = \frac{4S}{wd3} \times M \times \frac{\text{Scale reading}}{100\ \phi}$$

Where: E = Stiffness in flexure, Psi.
S = Span length
w = Width
d = Thickness
M = Weight on pendulum
ϕ = Angle in radiahs

In other words, these are "secant moduli" defining a line from the origin to the point on the line at the particular angle of measurement.

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*To confirm that no permanent change was suffered by any samples due to previous testing, at least one specimen of each material previously unflexed was tested at $-60^{\circ}F.$ The resulting curves were very close to those obtained on three samples actually used.

Figures 1 - 6 show the actual curves of moment against angle, at each temperature, for the Gum sample. Figure 7 shows a plot of modulus against temperature at each angle, and Figure 8 shows a plot of modulus against angle as a function of the temperature. Figures 9 - 16 show the corresponding data for GR-S, and Figures 17 - 24 for Polyethylene.

Discussion

The reproduciblity of curves on three replicate specimens, in most cases, was good. Instead of averaging points to make a synthetic "average curve", one of the actual curves, usually the one lying between the extreme values, was used for calculating stiffness.

Instead of attempting to draw a tangent to the start of the curve, as required by ASTM D 747, it was considered more practical to assume a straight line from 0 to 10° and the first modulus is given for this angular deflection. Stiffness values at other angles, then, become slopes of lines connecting the origin with the particular angular point.

The gum stock is the only material in the group which gives anything like a straight line relationship over this range of strain down to -80°F. At this temperature, its behavior is a decided curve with a steadily decreasing "stiffness" with increasing angle.

GR-S shows a curved response at all temperatures, and at -80°F. it resembles the curve of polyethylene at room temperature, with a "stiffness" of 94,000 psi. at 10° to 19,000 at 80° angle.

Polyethylene shows a curved response at all temperatures, with a sharp knee in the curve at -80°F.

To confirm the non-linear response of these materials, tensile stress-strain curves, up to 100 percent elongation, were made on two specimens from each material, tested in the form of a parallel--sided strip $\frac{1}{4}$" wide and 4" between jaws.

Stress-strain curves were made at 73°F. at five (5) rates of jaw separation in an Instron Tensile Tester, with a strain gage dynamometer in which the full scale deflection was about .005". The rates of jaw separation were 0.1", 1.0", 5", 10", and 20" per minute.

Typical curves, plotted from the original data made at 5"/min. are shown in Figure 25.

Stiffness values were calculated from the stress-strain relationship at 2.5 percent elongation, comparable to the strain at small angular deflection on the Olsen, and are summarized in Table II.

TABLE II

Tensile Modulus*, psi, at 2.5 Percent Elongation, Instron, 73°F.

Rate of Extension in./min.	Rate of Extension percent/min.	Gum	GR-S	Polyethylene
0.1	2.5	207	830	22,900
1.0	25.0	215	860	26,100
5.0	125.0	203	1080	31,700
10.0	250.0	205	1100	Very high
20.0	500.0	204	1190	Very high

The line for gum has a very slight uniform curvature within the elongation measured (100 percent). The tensile modulus at 2.5 percent elongation shows no significant change over the range of pulling speeds.

The line for GR-S has a sharply curved initial portion; the curve becomes linear at about 50 percent elongation, and the slope of the curve at $2\frac{1}{2}$ percent elonga-

tion is more than double the slope of this linear portion.

In polyethylene, a sharp knee is observed at about 12 percent elongation where the load to continue elongation levels off and even drops below that attained at the yield point.

Stiffness shows steady increase with rate of extension for GR-S and polyethylene.

On GR-S, nearly comparable values of stiffness are obtained at 10° angle on the Olsen, and at 2.5 percent tensile elongation at 20"/min. on the Instron.

On polyethylene, all stiffness values on the Olsen in the region below the yield point are lower than those obtained on the tensile tests at 2.5 percent elongation. The Olsen stiffness at 10° compares closest with the tensile stiffness at about 3 percent elongation tested at the slowest speed of 0.1"/min.

The polyethylene showed steady rise in tensile stiffness up to 5"/min. The rate of dynamometer loading, when testing this relatively rigid material at 10" and 20"/min., was too high for accurate response of the recording potentiometer, the load indicated lagged behind the actual load and hence gave low values of stiffness. At these rates of pull, the portion of the stress-strain behavior before the yield point takes place in about 3/4 to 1 second. Since the recorder takes about $1\frac{1}{2}$ seconds to respond to full scale, obviously the recording of phenomena of shorter duration will lag behind the event to some degree. A calibration curve of the response of the recorder gave lines with slope undistingushable from the test curve, hence no numerical value of stiff-

ness can be estimated for these two rates.

Conclusions

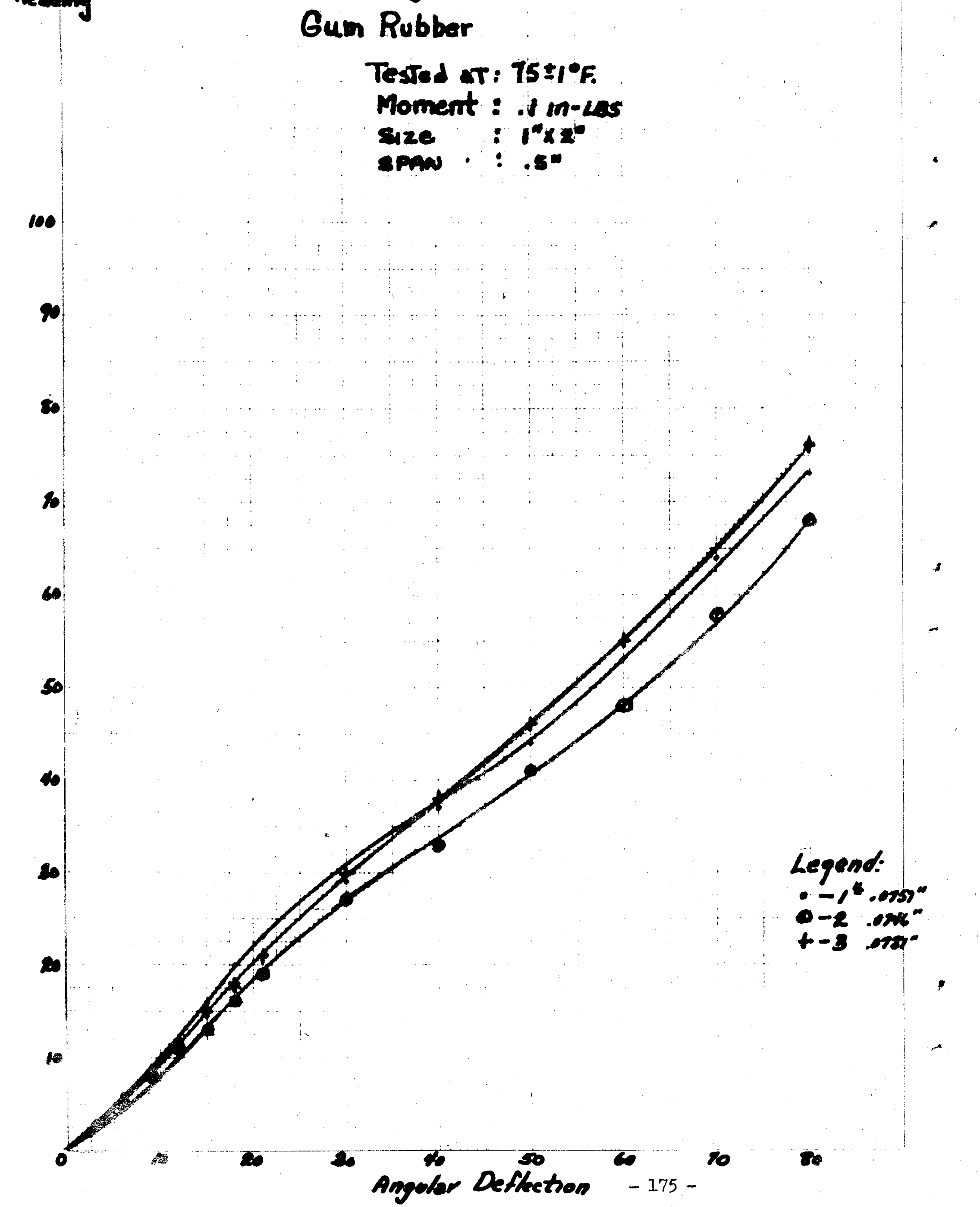
If we can assume that response to other applied forces such as shear, torsion, and compressive follows this same

*$\frac{\text{Stress}}{\text{Strain}}$ = Slope of stress-strain curve at 2.5 percent.

pattern of non-linearity at a fixed rate of deformation and, if it is as time-dependent as the tensile behavior indicates (and there is little evidence to the contrary), it becomes clear that there is no single index of "stiffness" that will characterize materials of this type. If "stiffness" per se has any inherent value, it will have to be arbitrarily defined at a definite strain (not stress) with an arbitrary limitation of time during which the straining occurs, and with the clear understanding that it cannot be extrapolated to cover other strains and other orders of straining rate.

This brings up the question of the validity of "stiffness" defined in any arbitrary manner as a measure of the combination of physical properties which make rubber useful, which is its nearly-elastic behavior over a wide range of strain. If we can establish that it loses this property in proportion to the increase in "stiffness," then "stiffness" and its measurement is justified. If this correlation is not completely valid, it would be more desirable to measure the "rubberiness" by a more direct method and use some simpler index of response to force, like Durometer or other form of indentation device to measure stiffness.

Fig. 1


Load Scale Reading
Gum Rubber
Tested at: 75±1°F.
Moment : .1 in-LBS
Size : 1"x2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
• – 1# .0757"
⊙ – 2 .0746"
+ – 3 .0781"

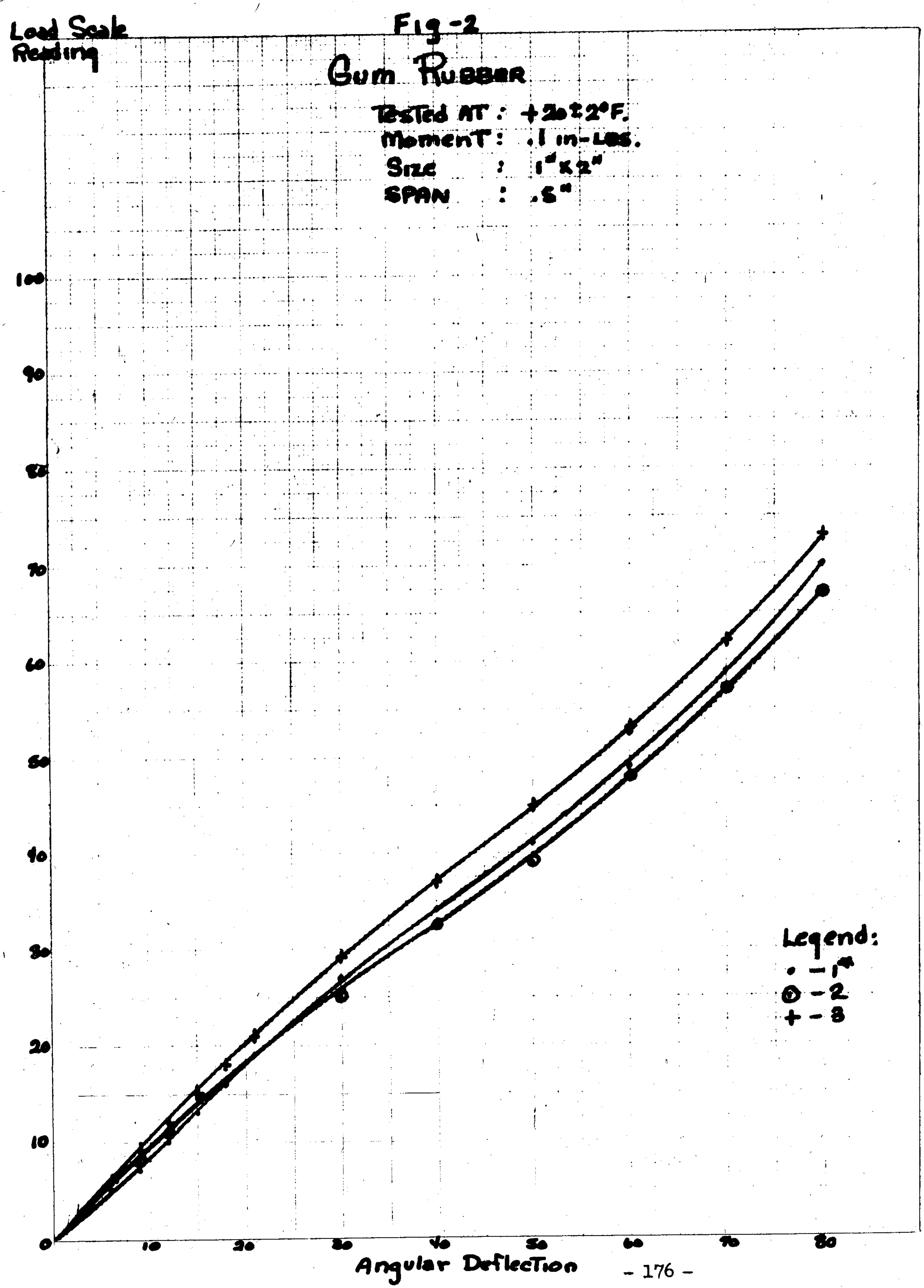
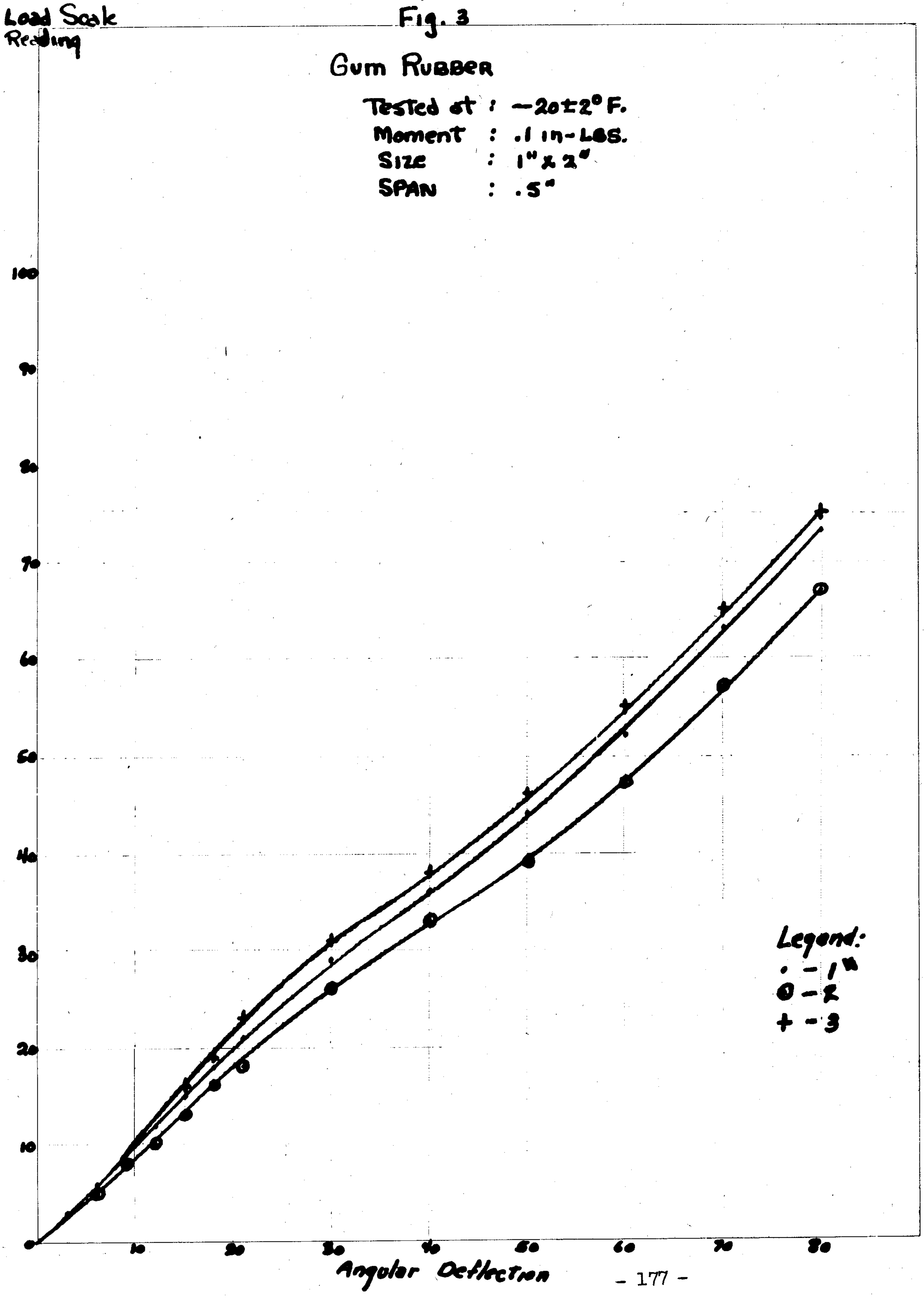

Load Scale Reading
Fig-2
Gum Rubber
Tested AT : +20±2°F.
Moment : .1 in-LBS.
Size : 1"x2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
• - 1st
⊙ - 2
+ - 3

Fig. 3


Load Scale Reading
Gum Rubber
Tested at : -20±2° F.
Moment : .1 in-LBS.
Size : 1" x 2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
10
20
30
40
50
60
70
80
Legend:
· - 1
O - 2
+ - 3
Angular Deflection

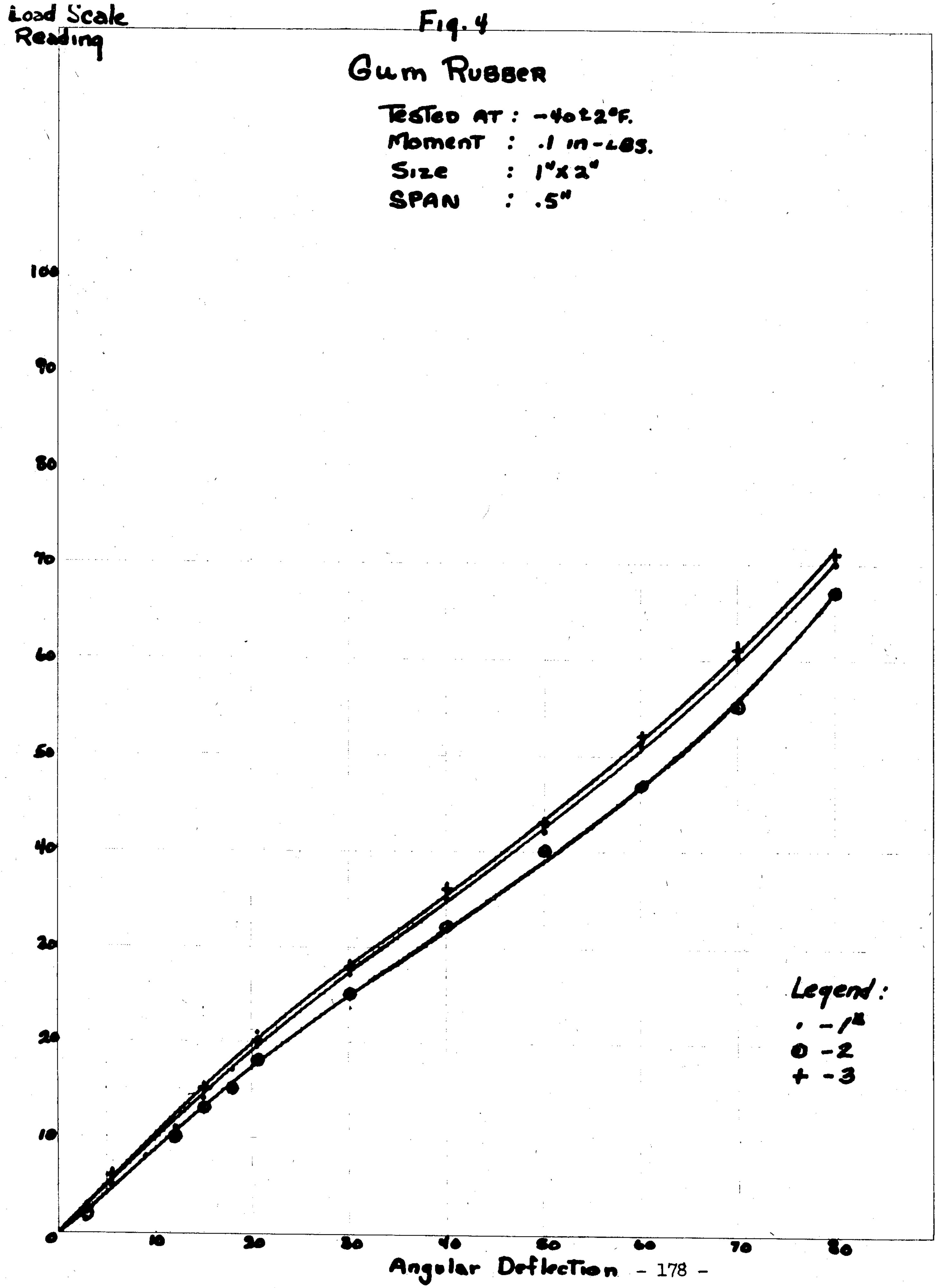

Load Scale Reading
Fig. 4
Gum Rubber
Tested at : -40±2°F.
Moment : .1 in-lbs.
Size : 1"x2"
Span : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
· - 1st
o - 2
+ - 3

Fig. 5

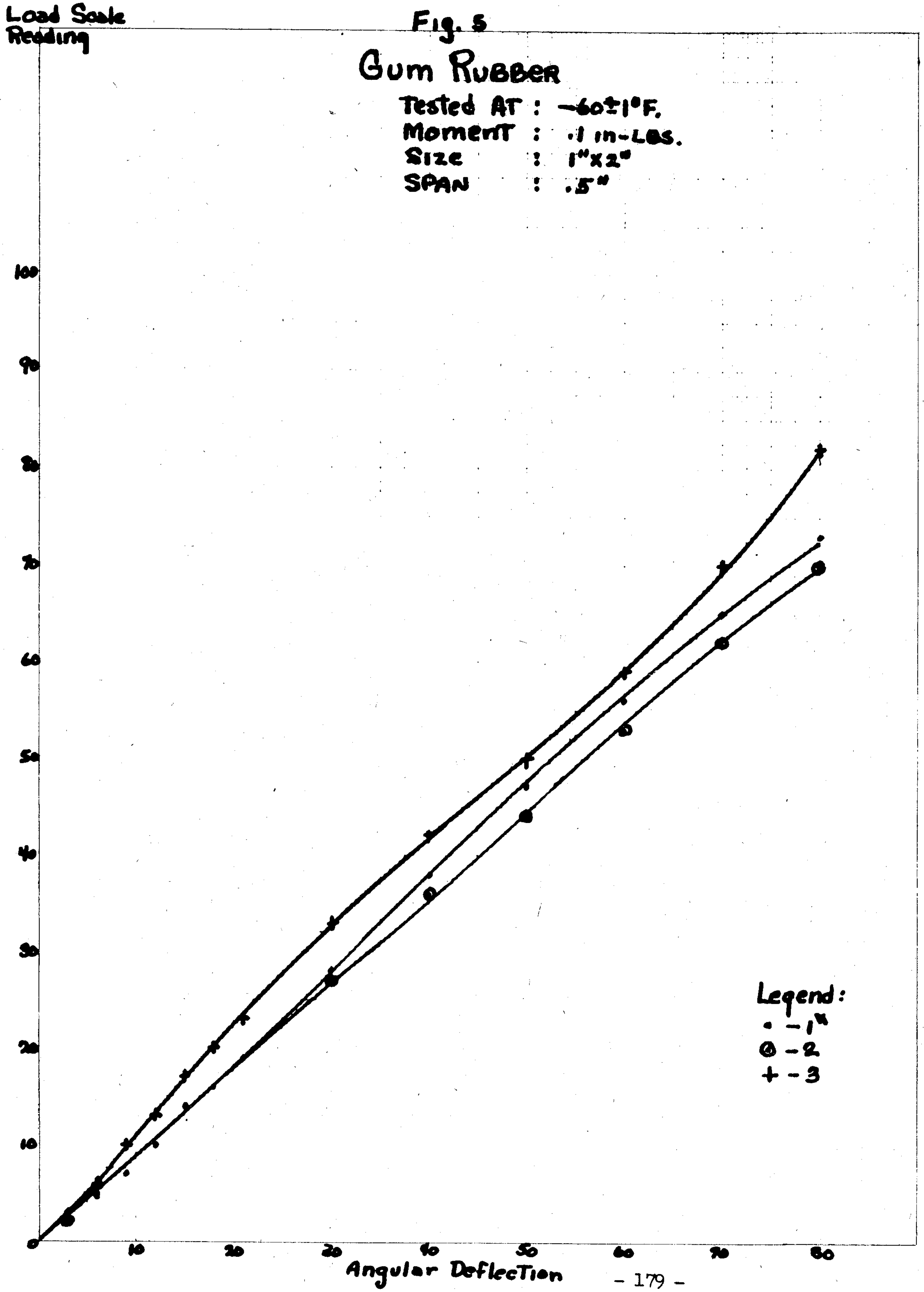

Load Scale Reading
Gum Rubber
Tested At : -60±1°F.
Moment : .1 in-Lbs.
Size : 1"x2"
Span : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
• - 1
⊙ - 2
+ - 3

Fig. 6

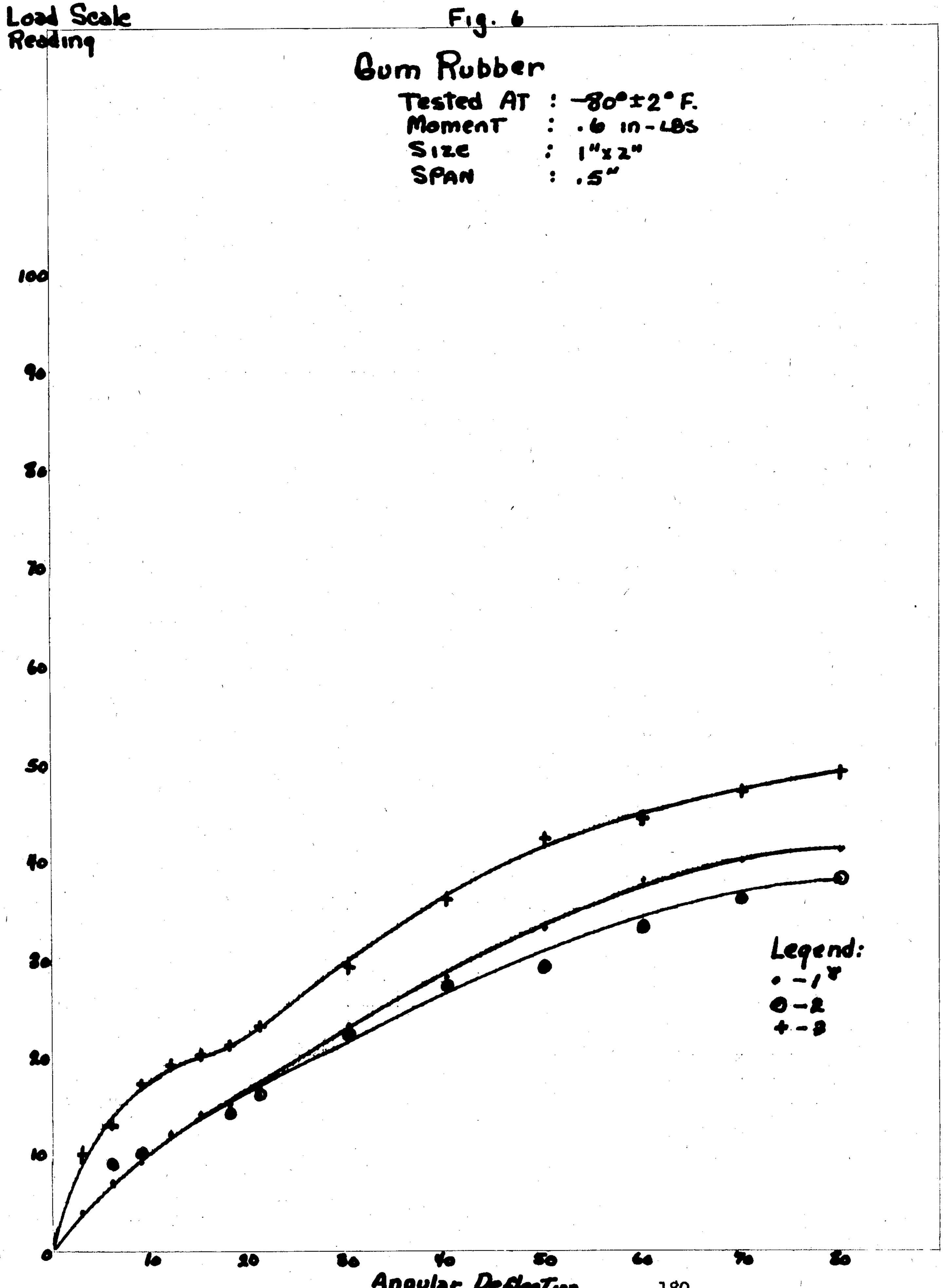

Load Scale Reading
Gum Rubber
Tested AT : -80°±2° F.
Moment : .6 in-LBS
Size : 1"x 2"
Span : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Legend:
• - 1
○ - 2
+ - 3
Angular Deflection

Fig. 7

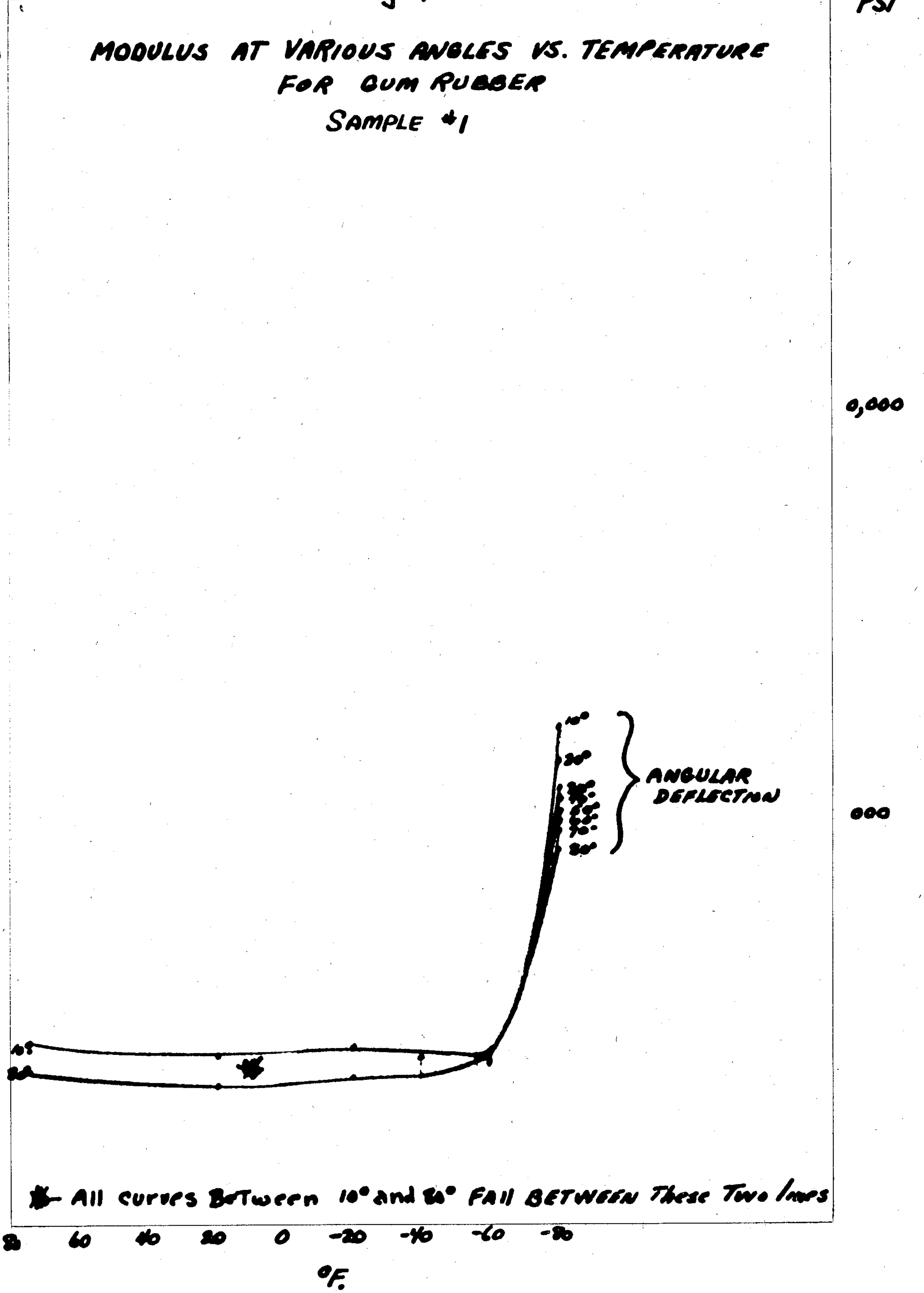

MODULUS AT VARIOUS ANGLES VS. TEMPERATURE
FOR GUM RUBBER
SAMPLE #1
MODULUS
PSI
0,000
000
ANGULAR
DEFLECTION
10°
30°
80°
10°
80°
* All curves Between 10° and 80° FALL BETWEEN These Two lines
80
60
40
20
0
-20
-40
-60
-80
°F.

Fig. 8

MODULUS AT VARIOUS TEMPERATURES VS. ANGULAR DEFLECTION
FOR. GUM RUBBER
SAMPLE #1

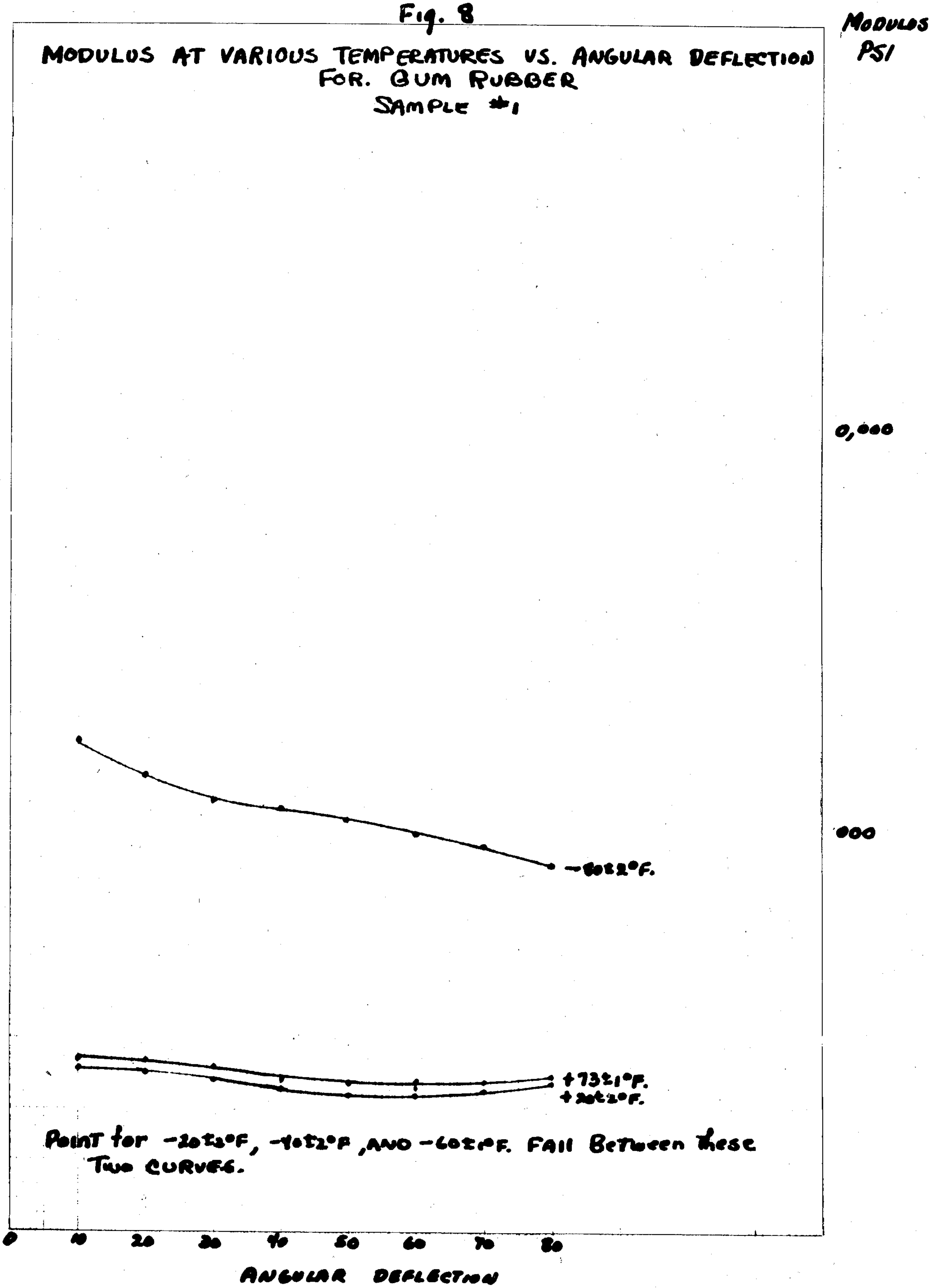


Fig. 9

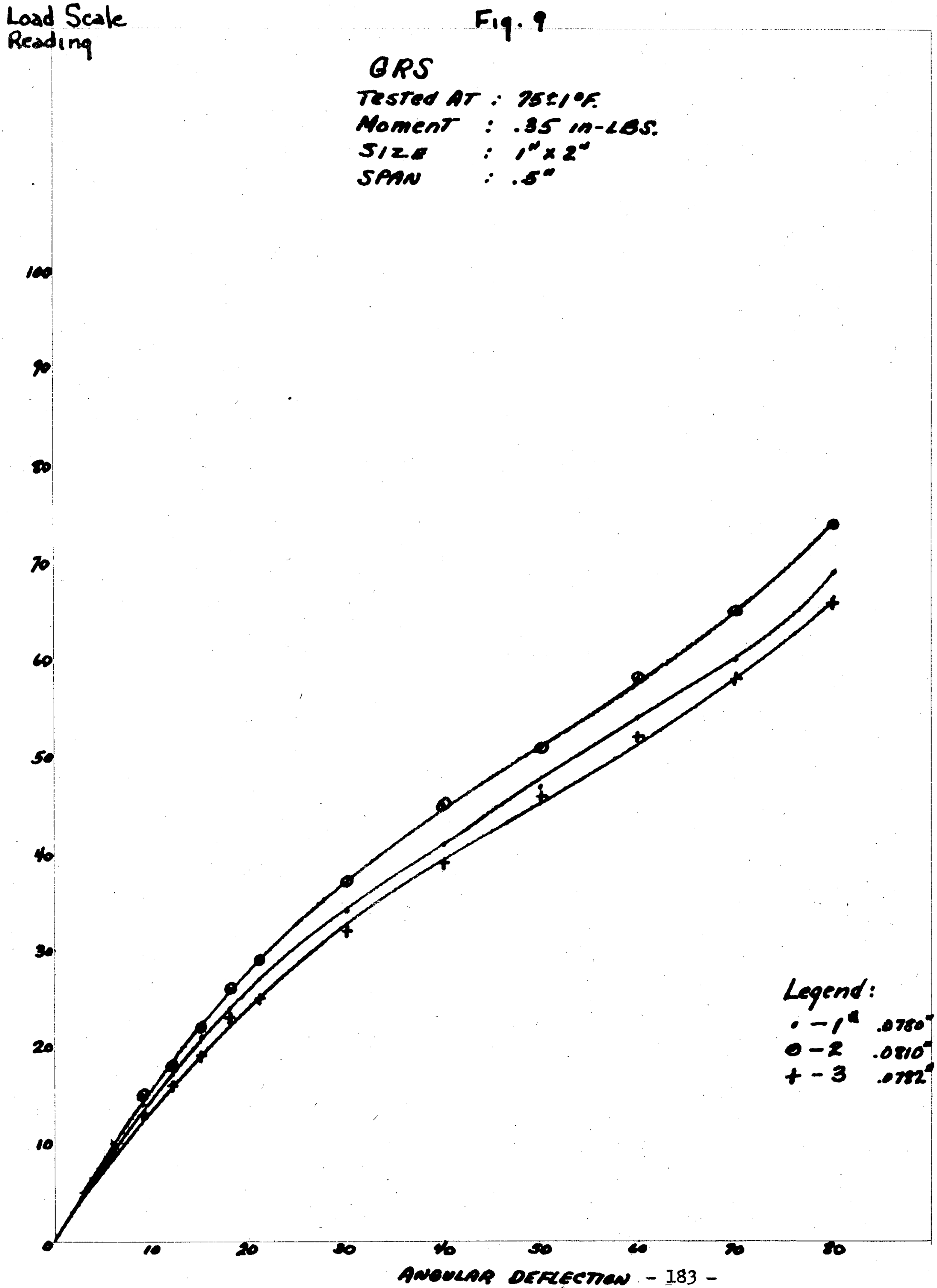
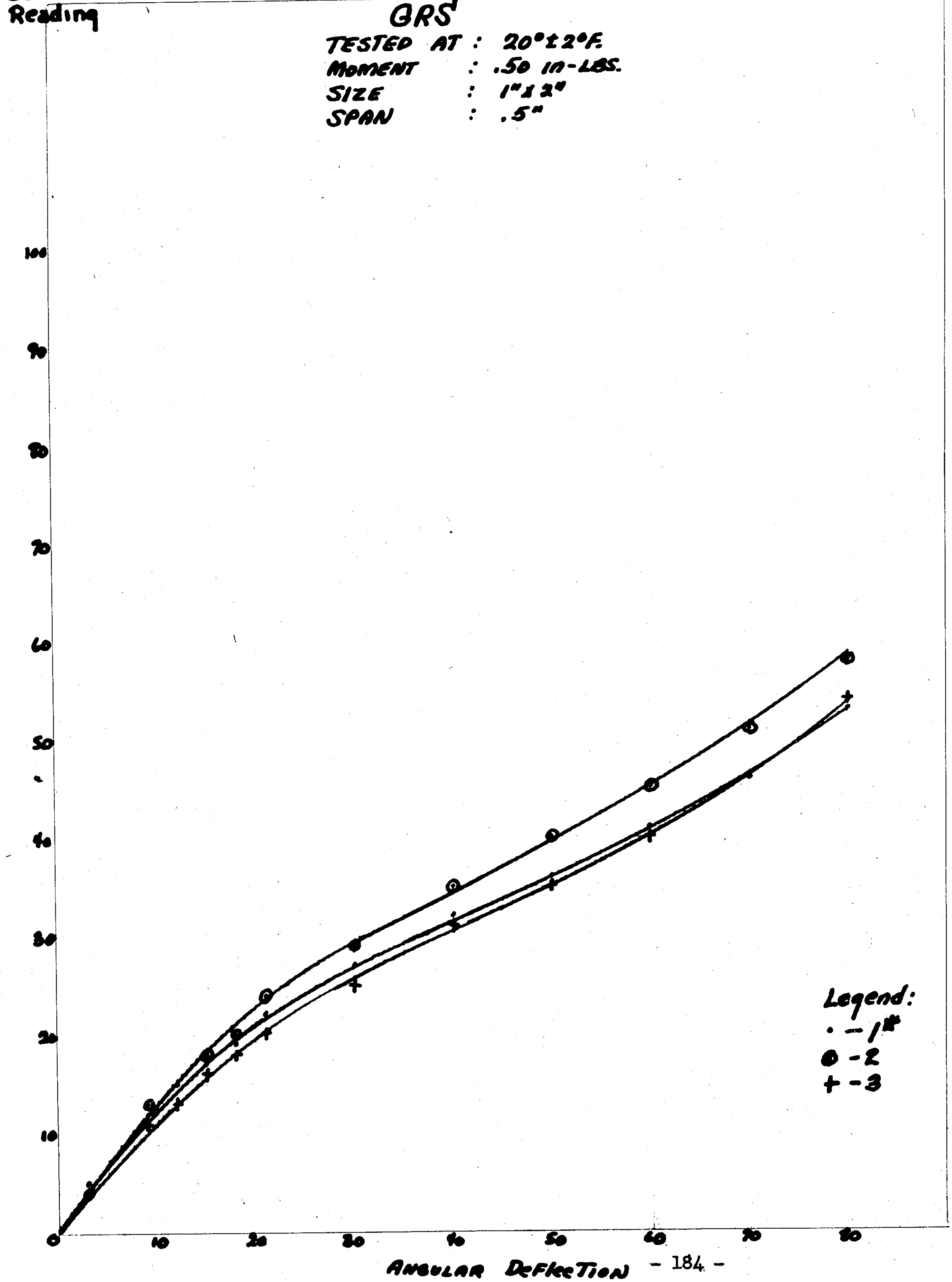
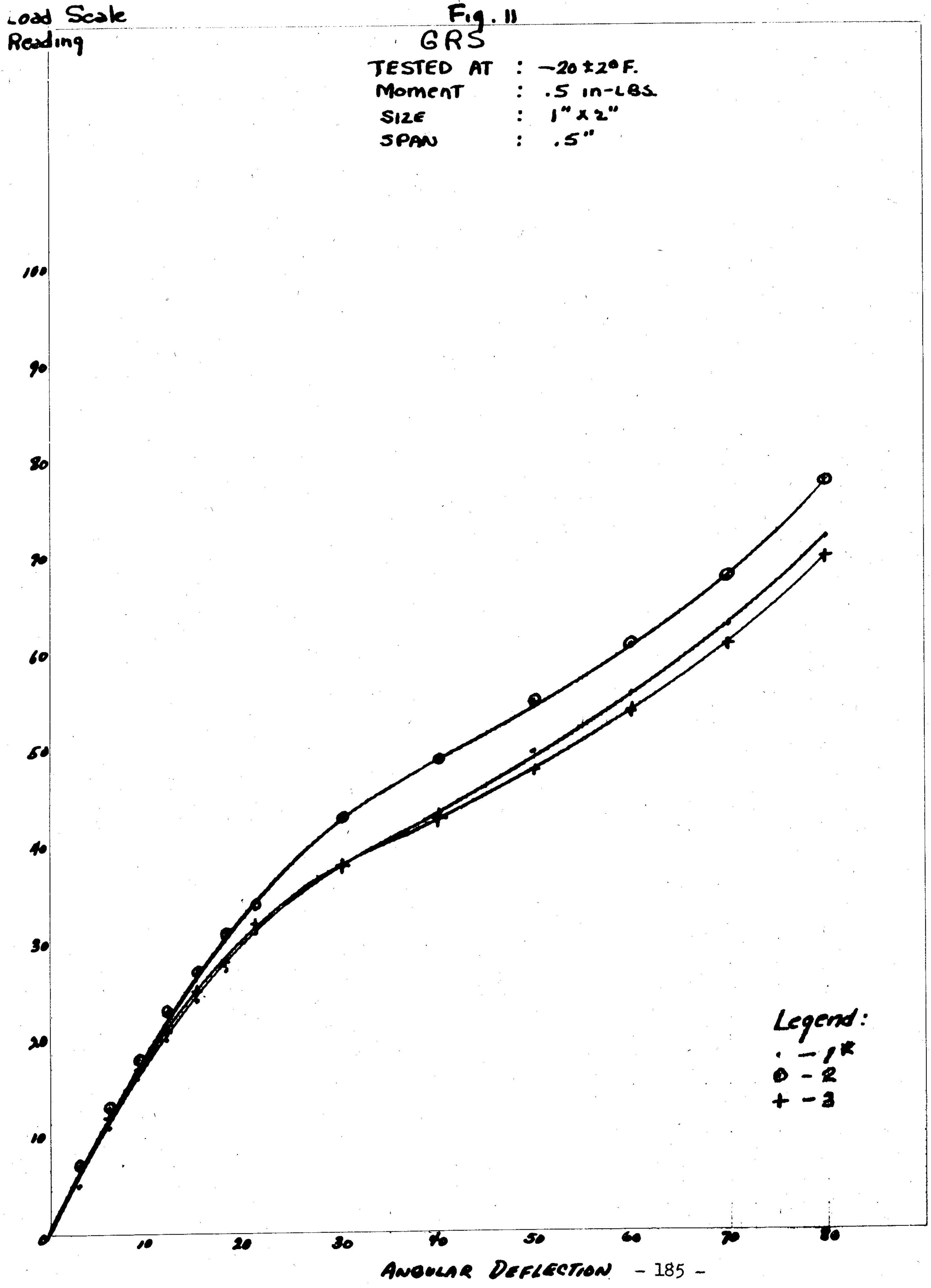

Load Scale Reading
GRS
Tested At : 75±1°F.
Moment : .35 in-LBS.
Size : 1" x 2"
Span : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
• - 1st .0780"
⊖ - 2 .0810"
+ - 3 .0782"

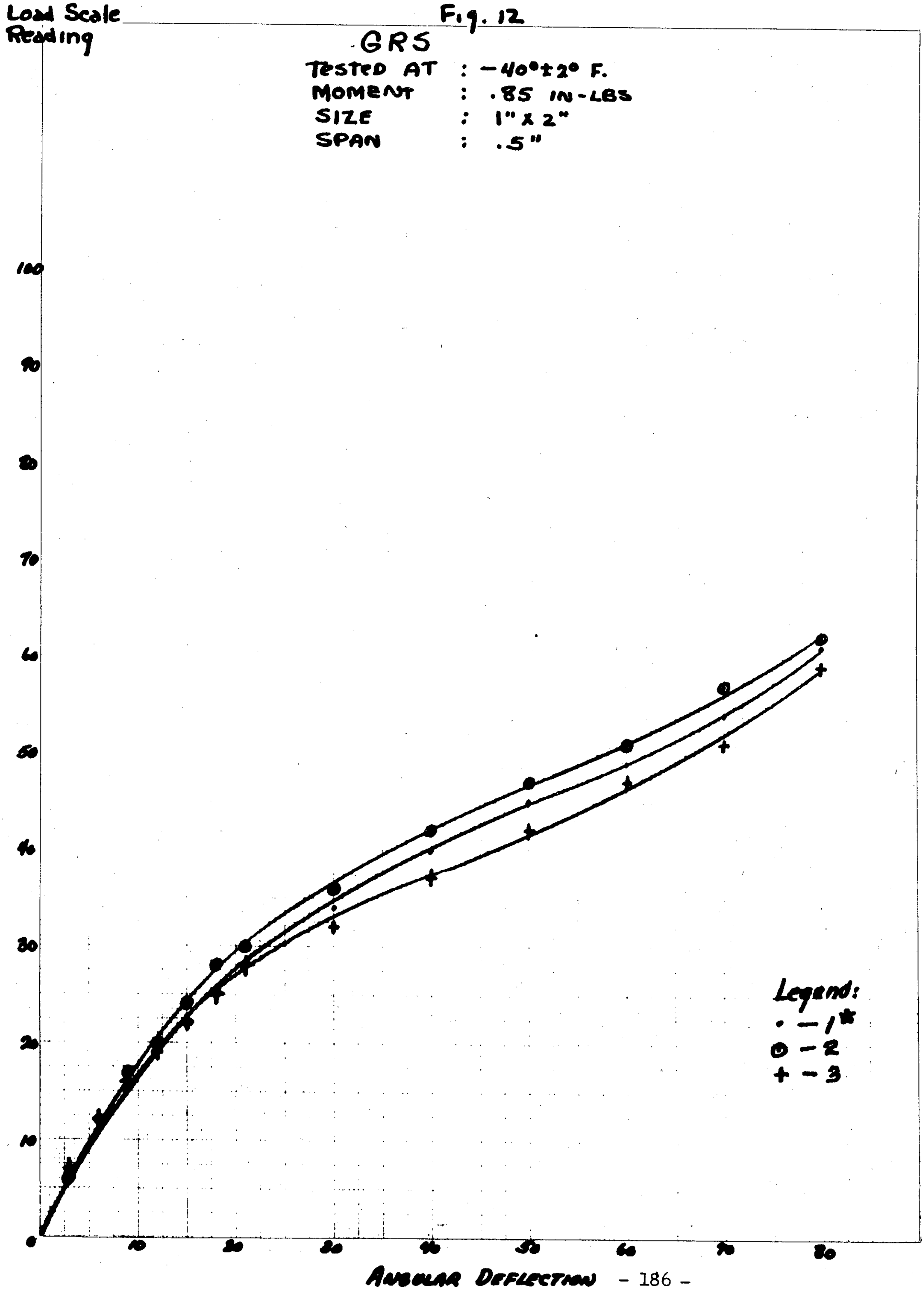
Fig. 10



Load Scale Reading
GRS
TESTED AT : 20°±2°F.
MOMENT : .50 IN-LBS.
SIZE : 1"x2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Legend:
• – 1st
⊙ – 2
+ – 3
ANGULAR DEFLECTION

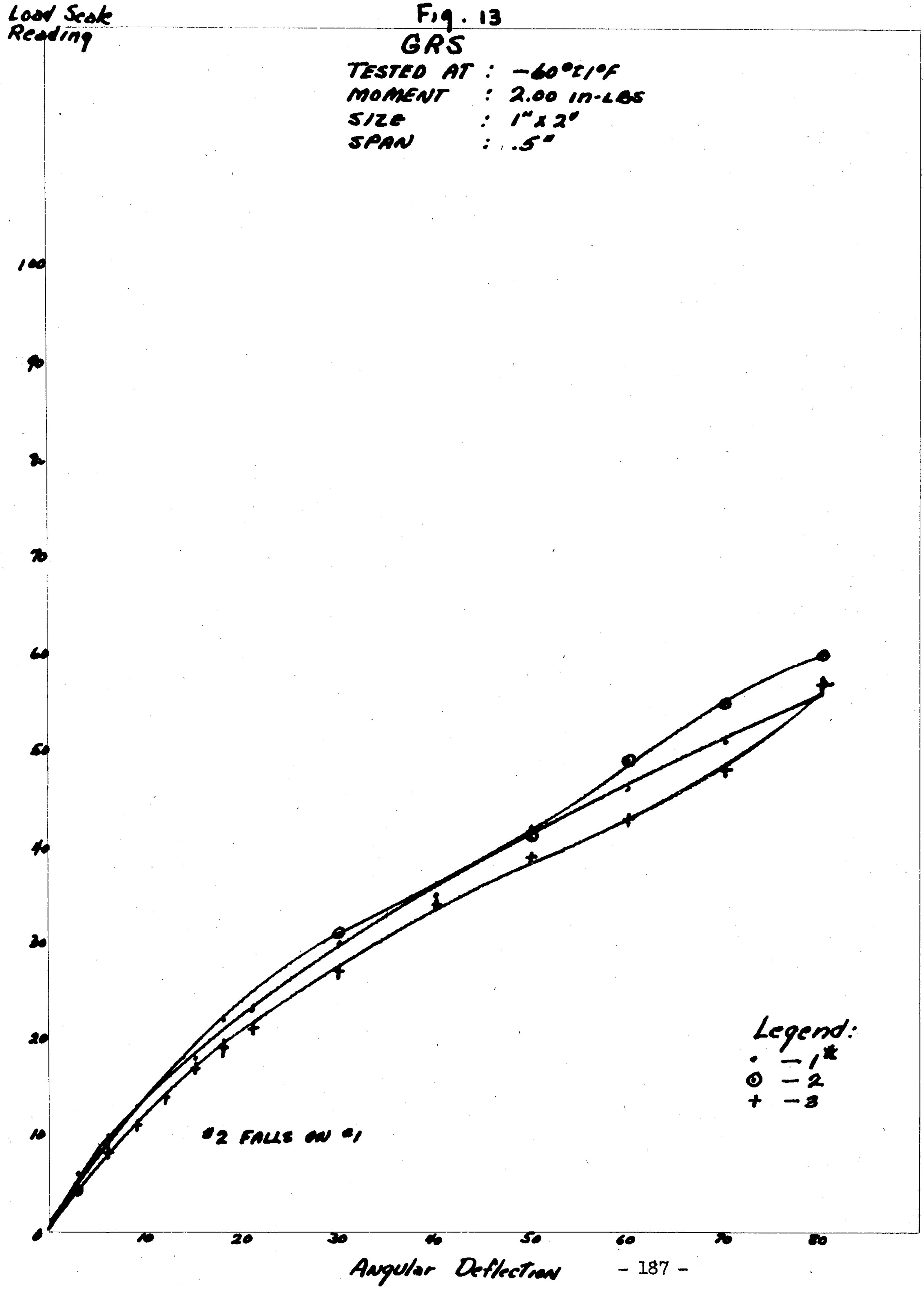
Fig. 11


Load Scale Reading
GRS
TESTED AT : -20 ± 2° F.
Moment : .5 in-LBS.
SIZE : 1" x 2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Legend:
· - 1
⊙ - 2
+ - 3
ANGULAR DEFLECTION



Load Scale Reading
Fig. 12
GRS
TESTED AT : −40°±2° F.
MOMENT : .85 IN-LBS
SIZE : 1" x 2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Legend:
• — 1*
⊙ — 2
+ — 3
ANGULAR DEFLECTION

Fig. 13


Load Scale Reading
GRS
TESTED AT : −60°±1°F
MOMENT : 2.00 in-LBS
SIZE : 1" x 2"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
#2 FALLS ON #1
Legend:
· — 1*
⊙ — 2
+ — 3
Angular Deflection

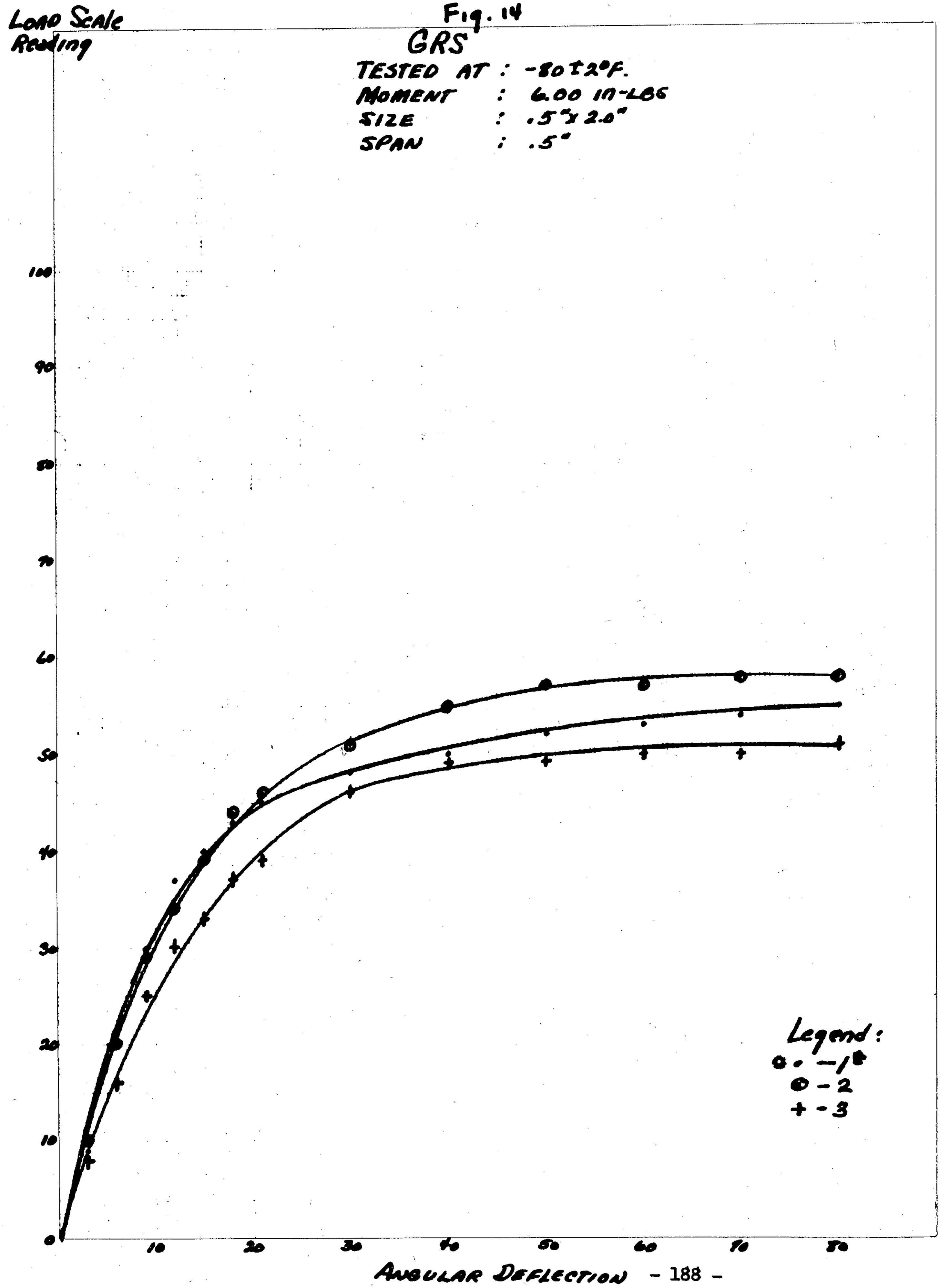
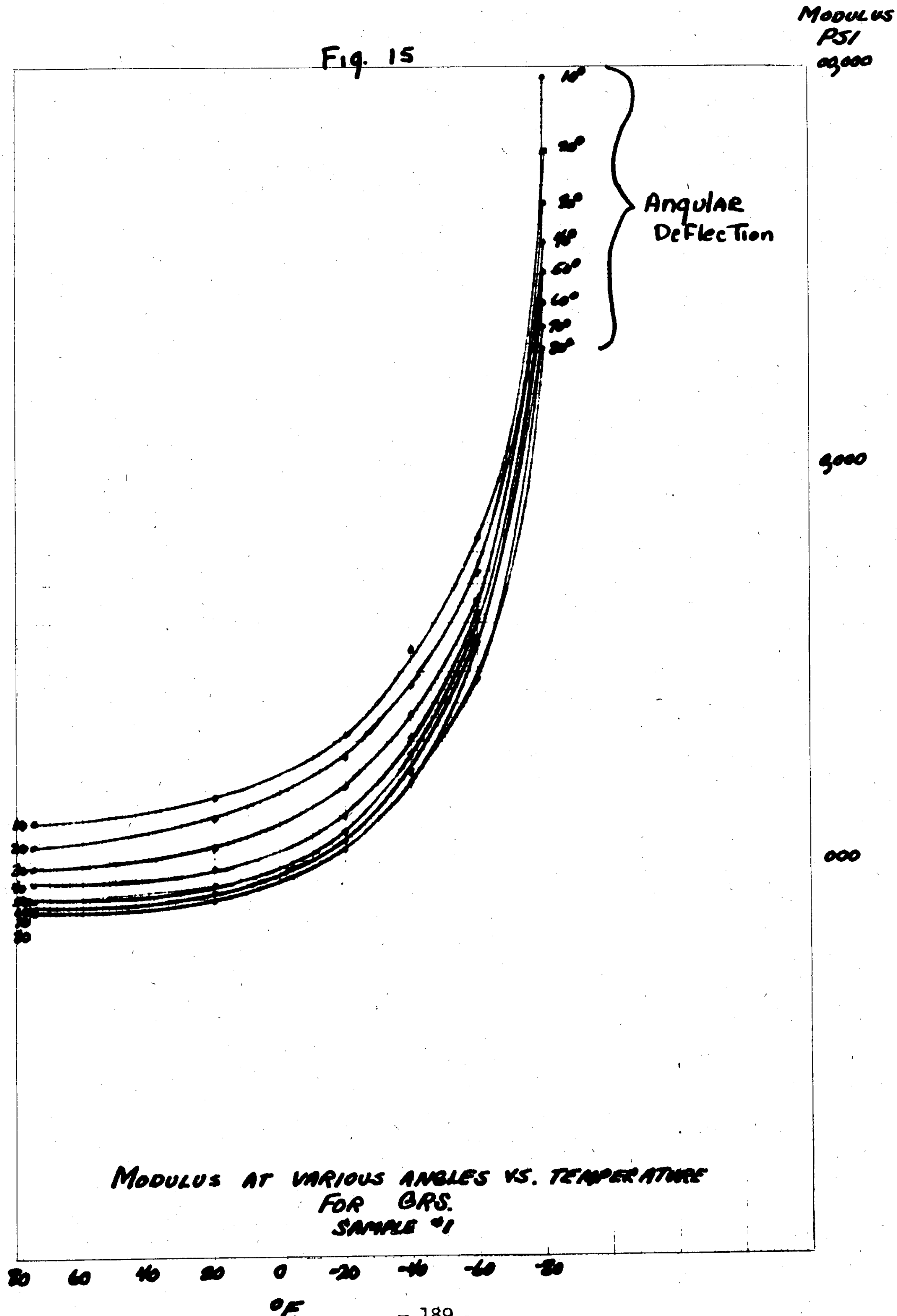
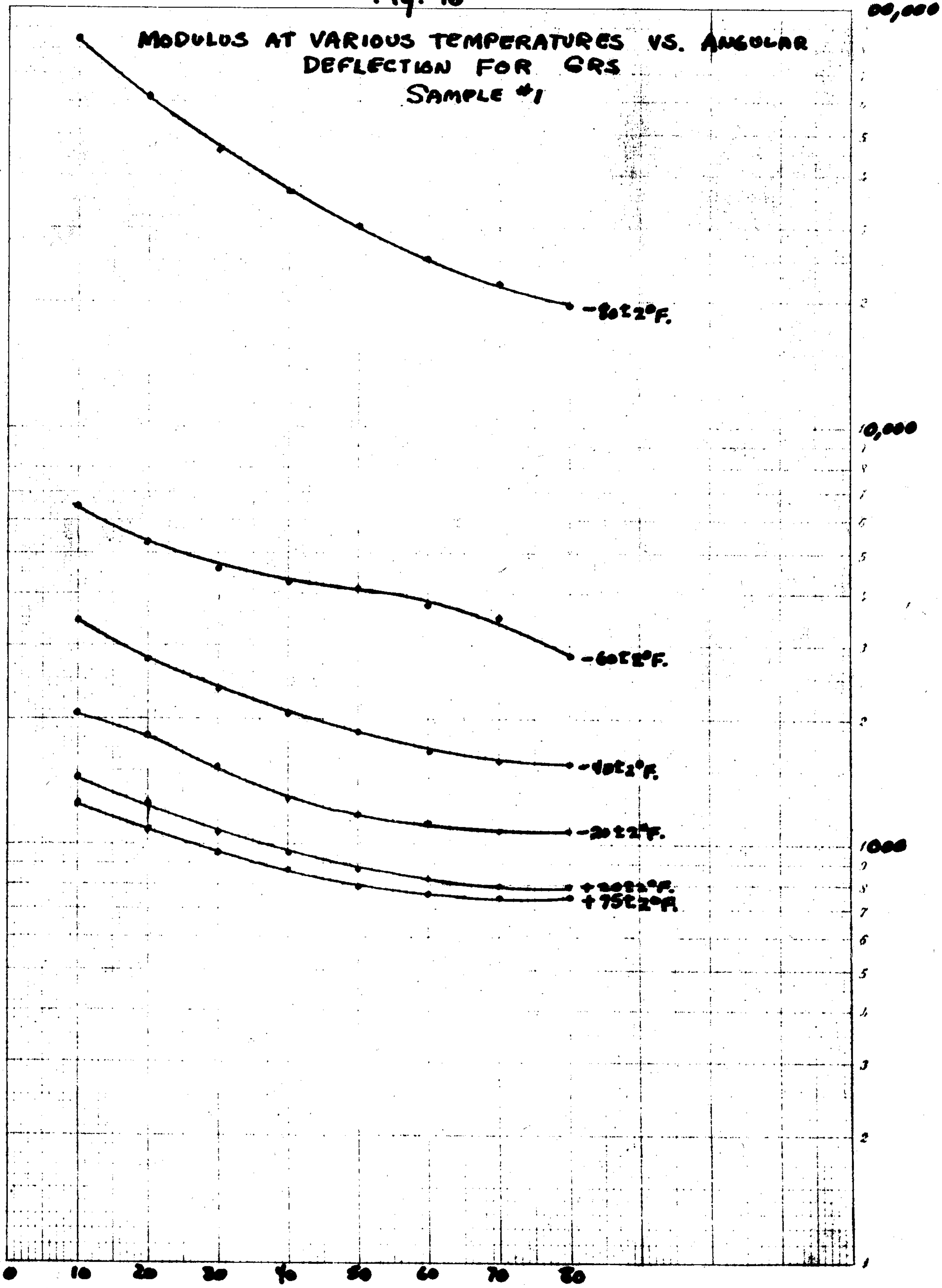

Load Scale Reading
Fig. 14
GRS
TESTED AT : -80 ± 2°F.
MOMENT : 6.00 IN-LBS
SIZE : .5" x 2.0"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
ANGULAR DEFLECTION
Legend:
• - 1
⊙ - 2
+ - 3

Fig. 15



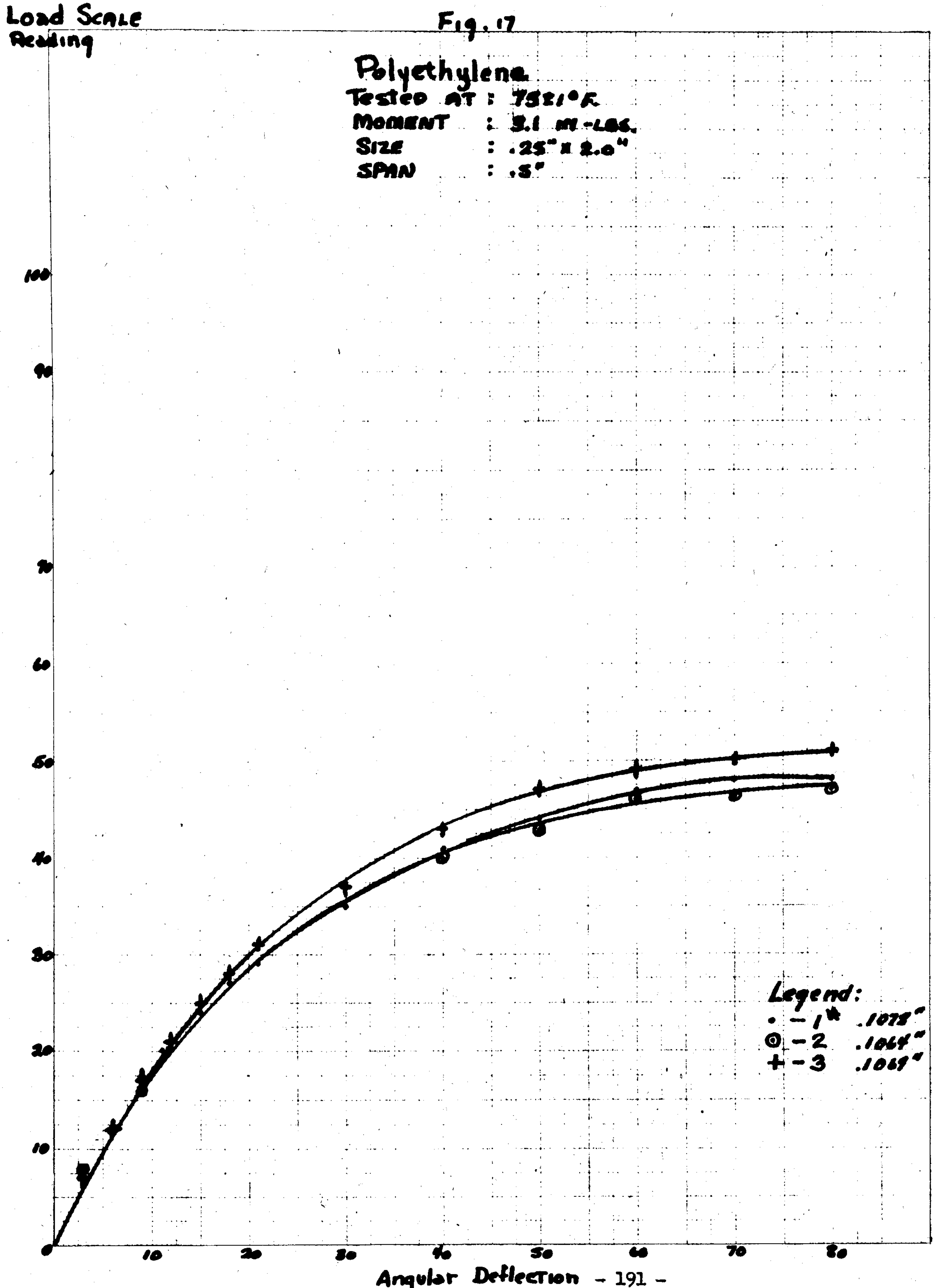
MODULUS AT VARIOUS ANGLES VS. TEMPERATURE FOR GRS. SAMPLE #1

Fig. 16



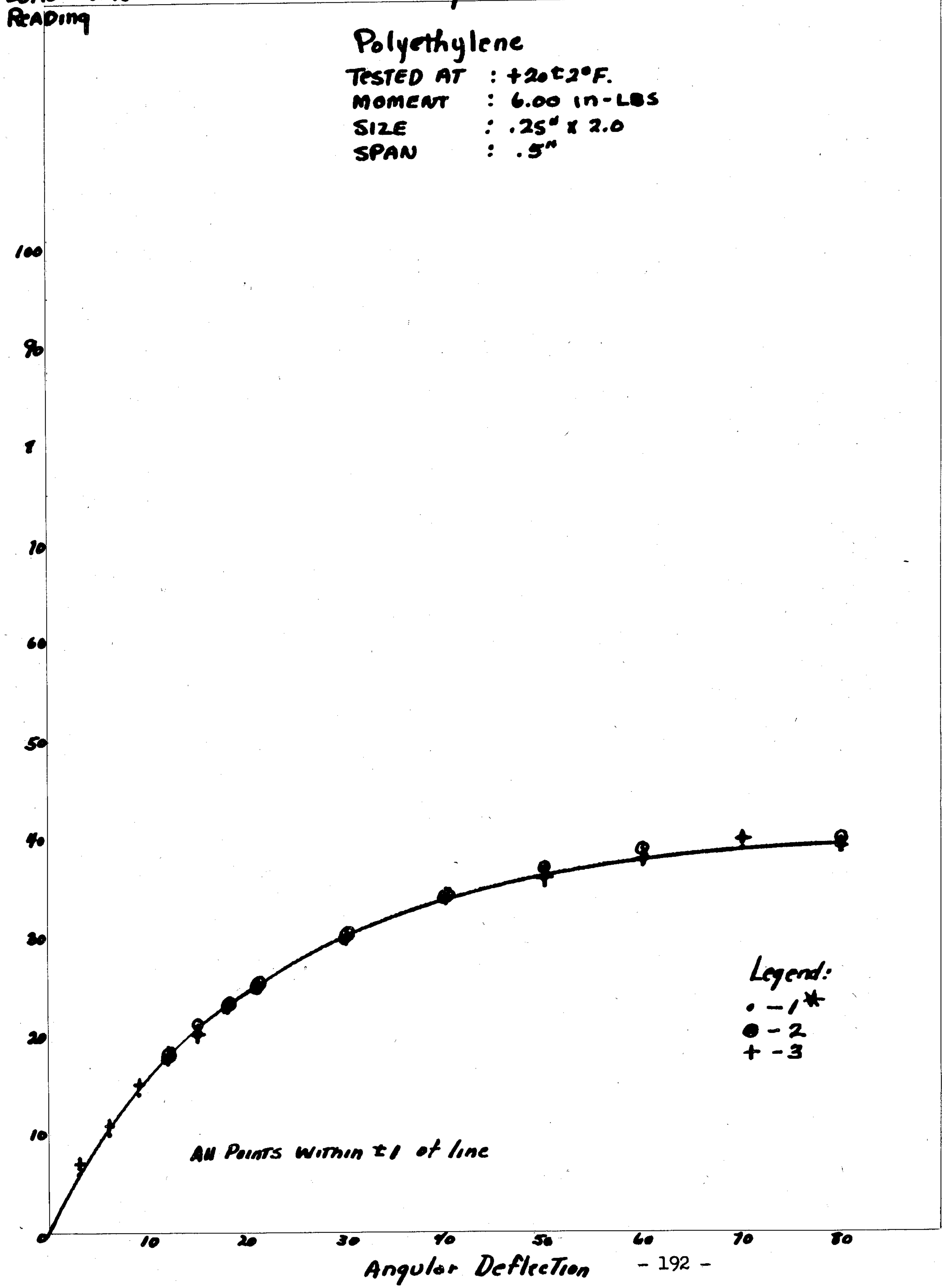
MODULUS AT VARIOUS TEMPERATURES VS. ANGULAR DEFLECTION FOR GRS
SAMPLE #1
MODULUS PSI
100,000
10,000
1000
1
2
3
4
5
6
7
8
9
-80±2°F.
-60±2°F.
-40±2°F.
-20±2°F.
+20±2°F.
+75±2°F.
0
10
20
30
40
50
60
70
80
Angular Deflection

Fig. 17



Load Scale Reading
Polyethylene
Tested at : 75±1°F
Moment : 3.1 in-lbs.
Size : .25" x 2.0"
Span : .5"
100
90
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Legend:
• - 1st .1078"
⊙ - 2 .1064"
+ - 3 .1069"
Angular Deflection

Fig. 18



LOAD Scale Reading
Polyethylene
TESTED AT : +20±2°F.
MOMENT : 6.00 in-LBS
SIZE : .25" x 2.0
SPAN : .5"
100
90
8
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
All Points within ±1 of line
Legend:
• – 1*
● – 2
+ – 3

Fig. 19

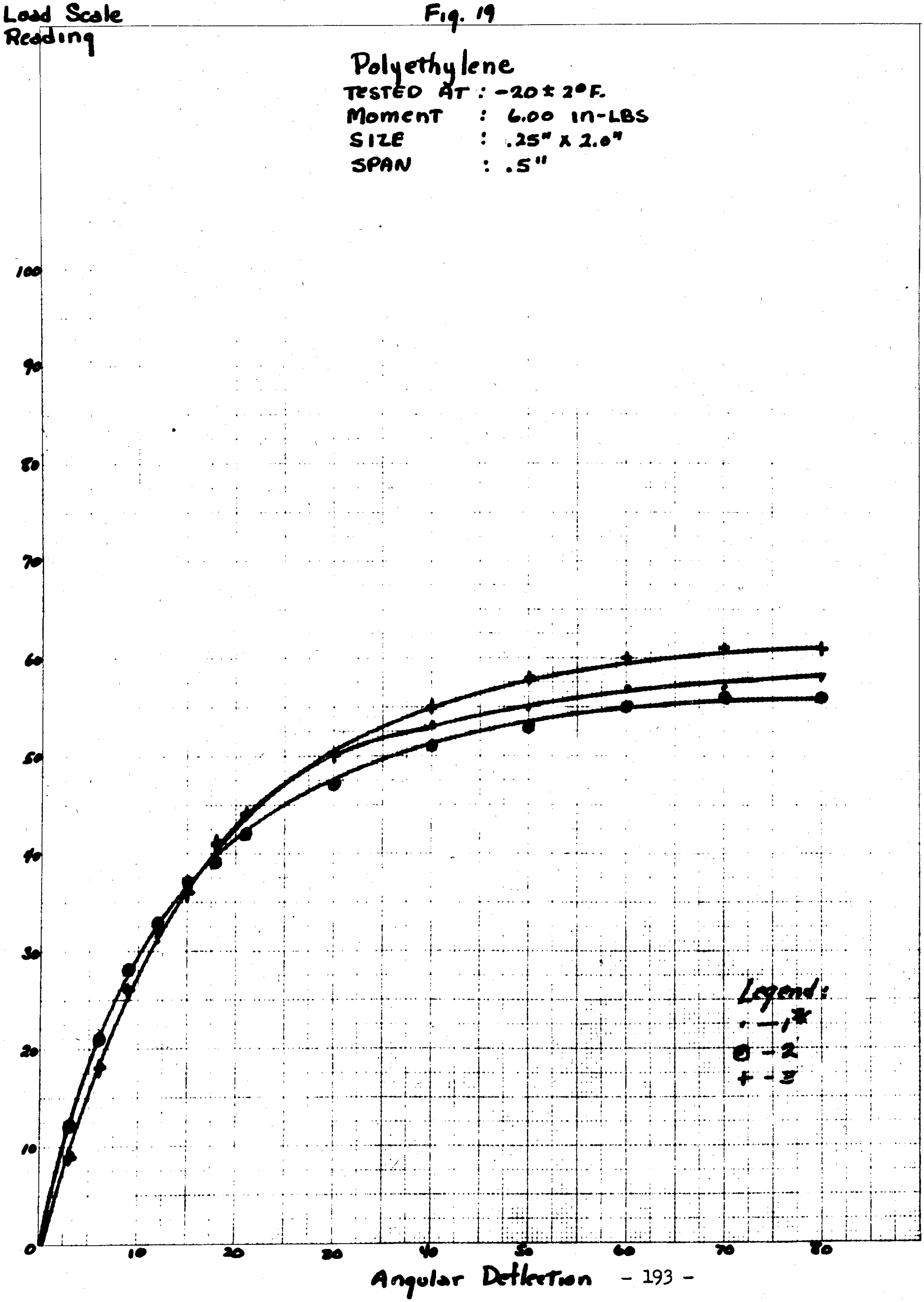
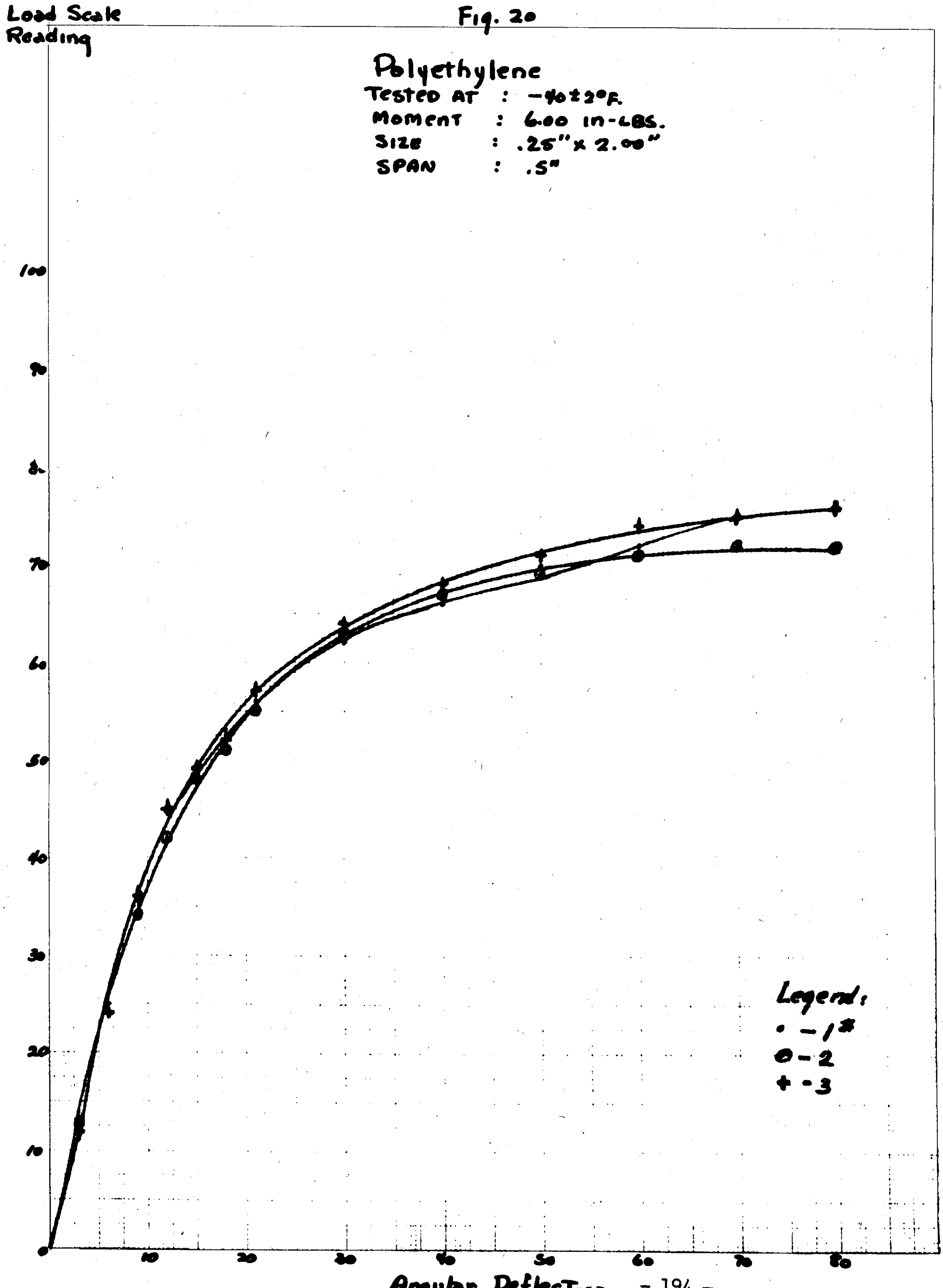
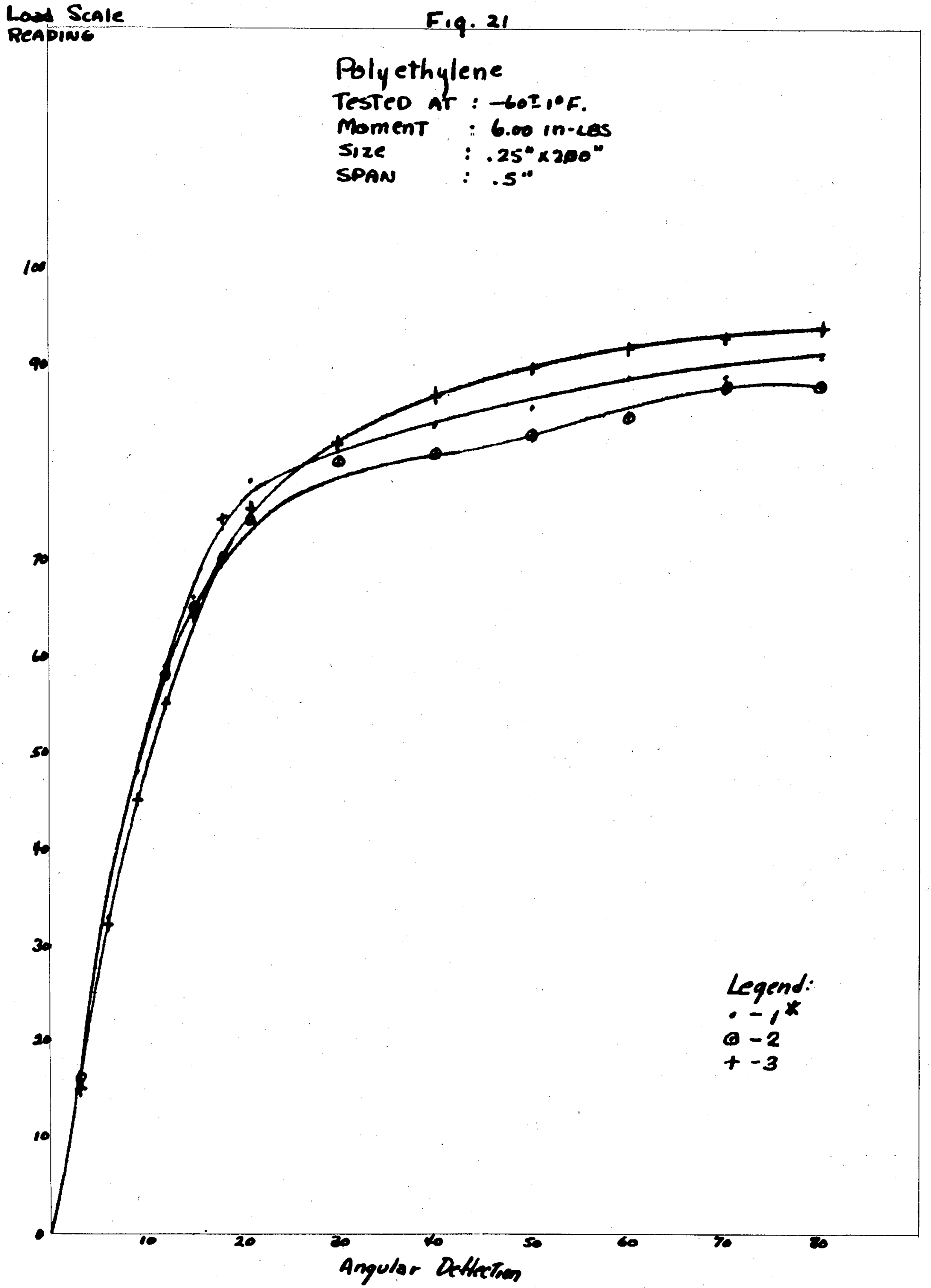

Load Scale Reading
Polyethylene
TESTED AT : -20 ± 2° F.
MomenT : 6.00 in-LBS
SIZE : .25" x 2.0"
SPAN : .5"
Legend:
• — 1
● - 2
+ - 3
Angular Deflection
0
10
20
30
40
50
60
70
80
90
100

Fig. 20


Load Scale Reading
Polyethylene
TESTED AT : -40±2°F.
MOMENT : 6.00 IN-LBS.
SIZE : .25" x 2.00"
SPAN : .5"
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
• - 1
⊖ - 2
+ - 3



Load Scale
Reading
Polyethylene
Tested at : −60 ± 1°F.
Moment : 6.00 in-lbs
Size : .25" x 2.00"
Span : .5"
100
90
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Legend:
• − 1*
⊙ − 2
+ − 3
Angular Deflection

Fig. 21

Fig. 22

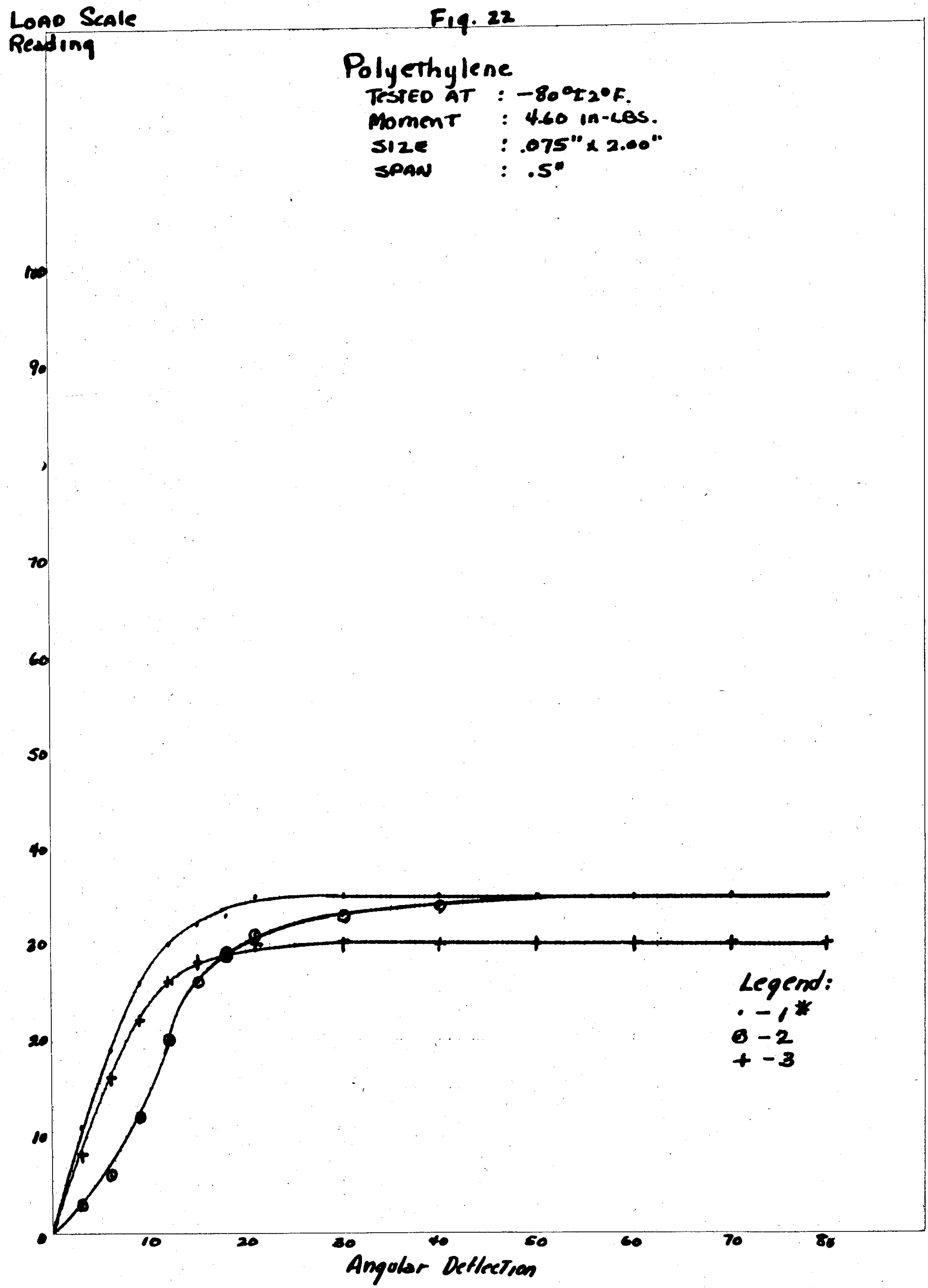

Load Scale Reading
Polyethylene
Tested at : −80°±2°F.
Moment : 4.60 in-lbs.
Size : .075" x 2.00"
Span : .5"
100
90
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
Angular Deflection
Legend:
· − 1*
⊖ − 2
+ − 3

Fig. 23

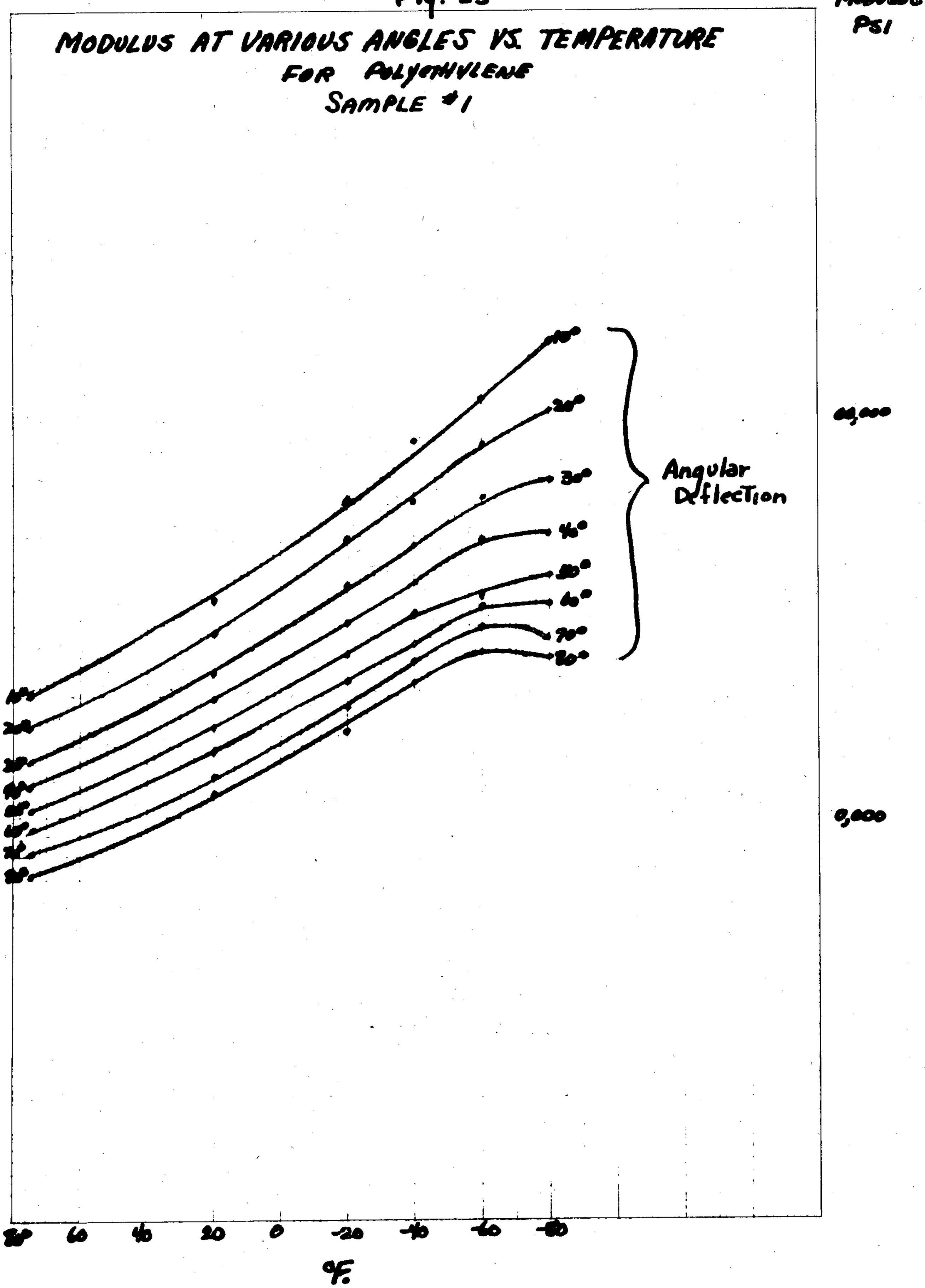
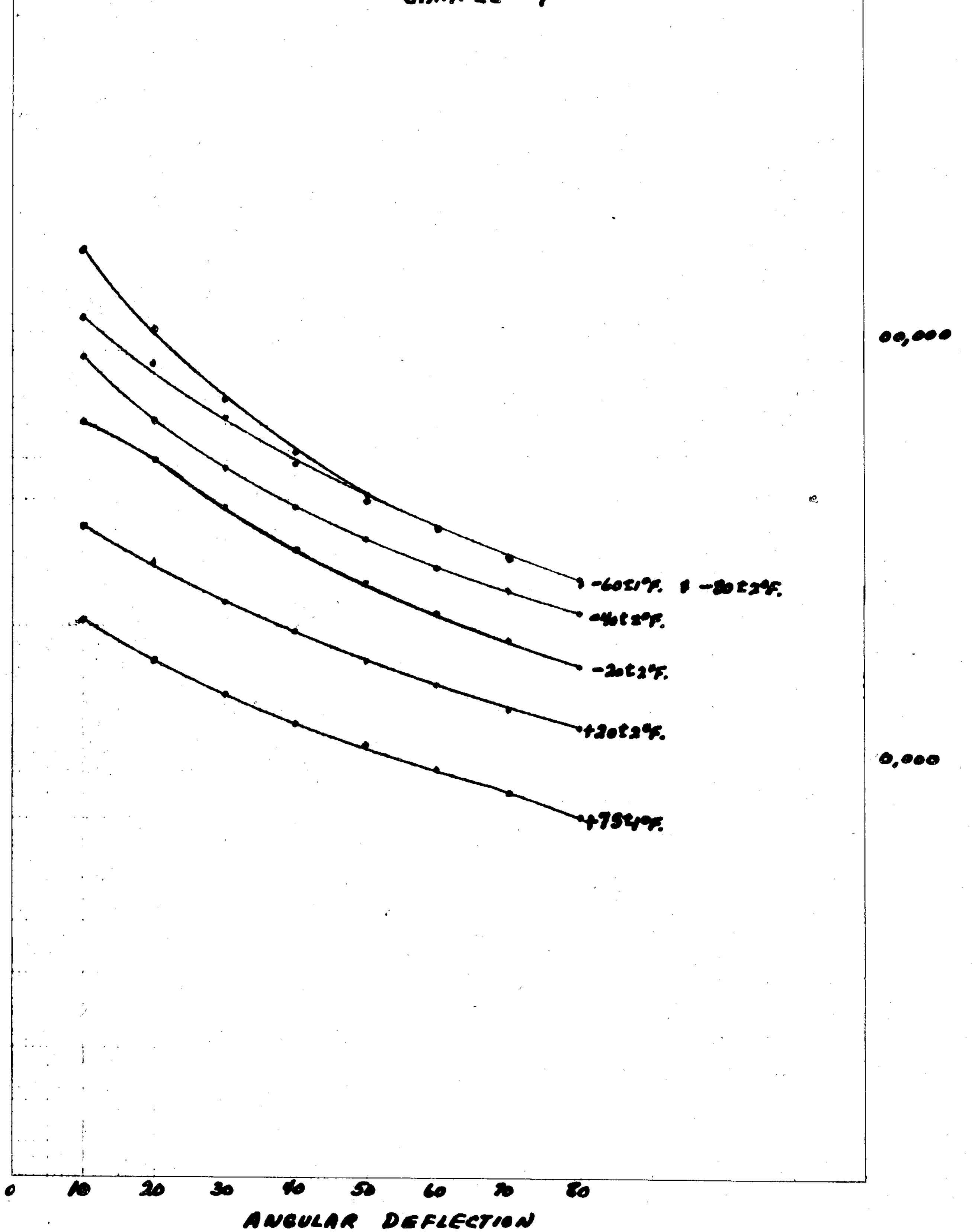

MODULUS AT VARIOUS ANGLES VS. TEMPERATURE
FOR POLYETHYLENE
SAMPLE #1
MODULUS
PSI
Angular
Deflection
10°
20°
30°
40°
50°
60°
70°
80°
80
60
40
20
0
-20
-40
-60
-80
°F.

Fig. 24

MODULUS AT VARIOUS TEMPERATURE VS. ANGULAR DEFLECTION
FOR Polyethylene
SAMPLE #1



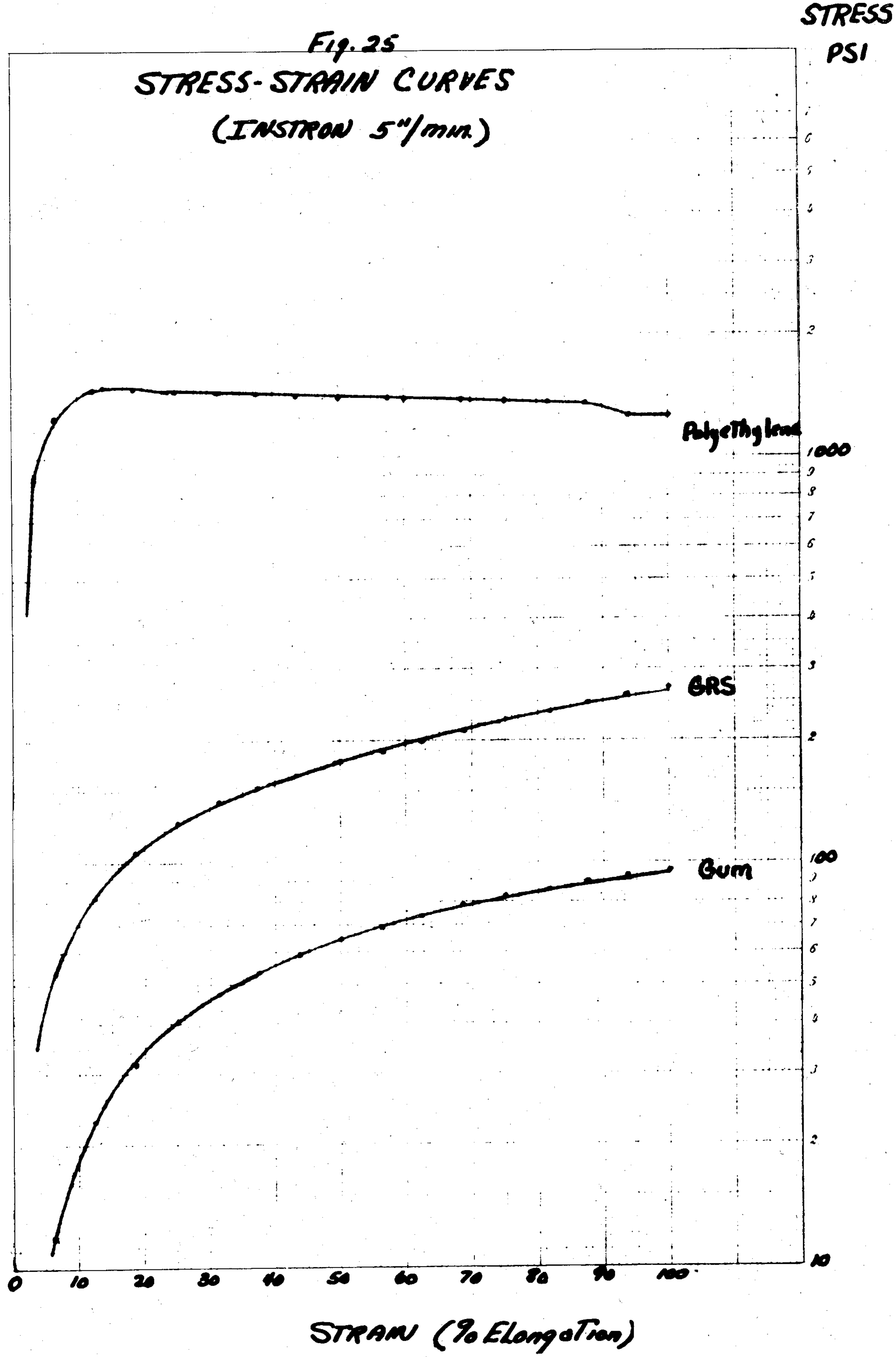


Fig. 25
STRESS-STRAIN CURVES
(INSTRON 5"/min.)
STRESS PSI
Polyethylene
GRS
Gum
1000
100
10
0
10
20
30
40
50
60
70
80
90
100
STRAIN (% Elongation)

LOW TEMPERATURE RUBBER TESTS

at the

Engineer Research and Development Laboratories

by

Philip Mitton and David M. French

1. As most of you realize, a rubber may fail at low temperatures: (a) because it is cooled below its First Order Transition Point and thereby becomes crystalline (b) because its viscosity is increased excessively because of decrease in kinetic energy and increase in forces of attraction between molecular sections, or (c) because it is cooled below its Second Order Transition Point where it becomes rigid or glassy due to cessation of rotation around chain bonds.

2. These three causes of failure are difficult to separate completely in tests of mechanical properties and for practical reasons it may be undesirable to do so. Probably all polymers crystalize to some extent; viscosity increases contribute to the failure even though crystallization is the ultimate cause of failure; and, in a sense, passing thru the Second Order Transition Point is an end point in the process of increasing viscosity. Because the temperature at which many elastomers become brittle approximates the Second Order Transition Point, many technologists have assumed erroneously that this is true for all elastomers. However, from a practical standpoint, the Brittle Point is more significant.

3. In general, however, at the Engineer Research and Development Laboratories (referred to hereinafter as ERDL)

we consider that we observe failures due to these three causes by the following tests:

a. The Tension Recovery Test and the Compression Recovery Test of crystallization,

b. The Torsional Stiffness Test of increase in viscosity,

c. A modification of the American Cyamamid Company Solenoid Test for brittleness.

4. All low temperature tests are performed and specimens are conditioned in the cold box shown in the first slide. This box has a test chamber three feet wide by two feet deep by three feet high which can be observed thru windows in the top and front. The second slide shows the interior with the low temperature test equipment. Tests are performed using electrically heated gloves fastened into arm holes in the front of the box as shown in the third slide. Specimens are always handled with tongs to avoid heating the specimens. The box is cooled by dry ice and is controlled to within plus or minus 2°F of the test temperature. In order to avoid the accumulation of snow in the air circulation system, cold air is withdrawn from the top of the dry-ice storage chamber and blown into the bottom of the test chamber. The air then passes from the top of the test chamber back to the bottom of the dry-ice chamber. It is still necessary to remove ice from the blades of the blower occasionally with steam.

5. Although rubber specimens must be conditioned at the test temperature for a period of time which may be a few minutes to a few months, in order to crystallize, viscosity changes and the second order change occurs as soon as the specimen is chilled to the test temperature. Nevertheless,

in our work, we are interested in the effect of crystallization on stiffness and brittleness; and also keep in mind that these tests will be used ultimately in specifications and therefore should be designed to indicate all changes in stiffness and brittleness. Therefore, not only the tension and compression recovery test specimens but also the stiffness and brittleness specimens are preconditioned for periods up to 28 days at the test temperature.

6. Crystallinity in rubber is commonly observed by following the changes in a property with time of storage at a low temperature. The property chosen may be its volume, its modulus of elasticity or some other property related to stiffness, or elastic recovery. The speed of crystal formation is dependent on the extent to which the specimen is deformed while the crystallites are formed, i.e. during storage at the low temperature. It is difficult to store and determine volume changes or stiffness changes in stretched specimens. However, elastic recovery can be conveniently measured by compression or tension recovery tests.

7. The compression recovery test is familiar to most of you but will be described briefly. (Slide 3). A standard compression set plug, 0.5-inch thick by 1.129 inches in diameter, is cut from a cured block and compressed in a clamping device. This device consists of two polished steel plates separated by spacers 0.35-inch thick and held together by two bolts. The plug is thus compressed to 70% of its initial thickness and is held under compression by the bolts. The specimen and clamp is conditioned for a pre-

determined time at the test temperature, ordinarily minus 65F. The clamp is then placed in a vise in the test chamber and opened with a wrench. The thickness of the specimen is determined exactly 10 seconds and again 30 minutes after release of the vise. The result is reported in terms of percent recovery. It has been found convenient to use a table showing the recovery corresponding to each possible initial and final thickness rather than make a calculation for each set of data.

8. The specimens and equipment used in the Tension Recovery Test are shown in the fourth slide. In this test, a standard T-50 specimen is stretched to 100% elongation and fixed in that position. The specimen and clamp are then conditioned for a predetermined time at the test temperature. The clamp is then released at that temperature while on a measuring board, supported at an angle of 15° to the vertical. The length of the specimen is determined 10 seconds and 30 minutes after release of the clamp and the percent tension recovery is calculated.

9. Both of these elastic recovery tests may be used to determine crystallizable elastomers. If recovery becomes smaller as the time of storage is increased, the decrease in recovery is attribute to crystallization. If on the other hand, recovery is not complete, even after 30 minutes, and is not decreased with increased storage under tension, the set is due to viscosity; or if no recovery is obtained even after 30 minutes, following a mere 30 minute conditioning period, the failure is probably due to the rubber being below its second Order Transition Temperature. In both of

these tests, the ten second recovery value is considered the more significant but cannot be used in specification work because results are too subject to error.

10. The compression recovery test is approaching obsolescence because it requires more time to indicate crystallization of the elastomer, requires a larger specimen (which may not be available), is more difficult to perform, and requires more storage space in the test chamber, yet the information obtained correlates very well with that obtained by the Tension Recovery Test. Nevertheless, this test has been recommended to the Air Force for inclusion in Military Specification MIL-R-5847A for "Rubber; Silicone, High and Low Temperature " in order to exclude crystallizable silicones. It was recommended that the 30 minute recovery, after storage for 7 days at minus 65 F should not be less than 80%.

11. The tension recovery test has been included in several Proposed Military Specifications for electric power cable prepared by the Corps. of Engineers. These specifications require that the tension recovery of the material shall be not less that 20% due to storage of the extended specimens at -65 F for 7 days when determination of Tension Recovery is made 30 minutes after the clamps have been relaxed. The same requirements have been included in ERDL procurement descriptions for experimental fire hose.

12. In addition to indicating crystallization, the tension recovery test has two very practical purposes. Since it correlates well with the results of the Com-

pression Recovery Test it may be, assumed that it gives a good indication of the suitability of rubbers for gaskets. In addition, we have found that this test indicates very well, whether rubbers are susceptible to what we have called the "Coiled Spring Effect" or not. I think this term can be explained best by reference to the fifth slide. In this picture we see two cables which were uncoiled from the rear of a truck at a low temperature. You will notice that one remained uncoiled, the other returned to a coiled spring. Obviously the second type is very objectionable. Similar effects have been observed in hose and items made of coated fabrics.

13. Recently we have tried the United States Rubber Co. retraction test. We believe that this test gives very important information regarding crystalization of polymers in much less time than the methods calling for prolonged storage at one temperature. However, a word of caution should be injected regarding the use of this instrument in specification work. Since crystallization and other changes in plasticizers due with time at low temperatures are not accelerated by stretching the specimen rubbers containing poor plasticizers are likely to be passed by this test.

14. The second order transition point of a polymer is determined most accurately from the change in slope in the volume - temperature curve. (By this I include the method of Drs. Wood and Work at the Bureau of Standards for measuring dimensional changes). However as mentioned above, we consider brittleness to be more important from a practical standpoint. This property is measured by means of the Am-

erican Cyanamid Company Solenoid Tester shown in the sixth slide. This instrument was selected at the suggestion of the Materials Laboratory of the New York Naval Shipyard. It has a stroke speed of 6.5-feet per second and bends the specimen through an arc of 90°. It has given satisfactory service but is probably no better than other machines such as the Bell Telephone Laboratory Tester. Poor correlation between various brittleness tests may be expected because the Brittle Points may not be assumed to be a linear function of the rate of bending. At ERDL, triplicate, T-50 specimens are stored for predetermined times in the cold box at the test temperature. These specimens are then tested in the Brittleness Tester which is also placed in the cold box for this purpose. In this way any effect that extended storage has on brittleness may be observed. The three specimens, joined together with cellophane tape before storing, are placed in the clamp of the tester by means of tongs. The clamp is returned to the test position. The specimens are bent thru an angle of 90 degrees by a solenoid activated bar. Much desirable academic information on brittleness has been left lacking because most rubbers become unserviceable, for other reasons, at temperatures well above their brittle points. In general, the brittleness test is used as a screening test, in which the stocks which pass are considered worthy of further consideration.

15. This test has been included in the above mentioned proposed power cable specifications and procurement description for experimental fire hose. In these, it is required that specimens shall withstand the test

without fracturing or cracking. It is required that the test be performed at -65 F on specimens which have been stored at that temperature continuously for 7 days.

16. The tendency of most rubbers to become stiff at low temperatures is one of their worse defects. This tendency has been measured at most laboratories by the use of methods which give a temperature-stiffness curve. Unfortunately, these tests are made ordinarily on specimens that have not been preconditioned at the low temperature. As a result they do not indicate the effects of polymer crystallization or changes in plasticizer with time. At ERDL it was decided that stiffness tests would be performed in the cold test box, on specimens which has been preconditioned at the test temperature (which is ordinarily minus 65°F) for predetermined periods of time.

17. The Gehman Torsional Stiffness Tester was selected for this work because it has no bearings to be effected by temperature changes. In this test, shown in the seventh slide, the rubber test strip is held rigidly by a clamp at the bottom and by a clamp which is also attached to one end of a standard wire at the top. In the test procedure the other end of the standard wire is rotated 180°. Means is provided for measuring the angle of twist (a, in the formula below) of the rubber specimen. This angle is determined 10 seconds after rotating the top end of the wire. The results of the Gehman test are calculated in the form of what we term the Torsional Stiffness Factor which is equal to $(\frac{180-a}{a})$. The ratio of the stiffness factor obtained on specimens at -65°F to that obtained at room temperature (73.5°F) is referred to

as the Torsional Stiffness Ratio.

18. It would be better perhaps to report the reciprocal of the Torsional Stiffness Ratio multiplied by 100 since the numerical difference between successive measurements on the same sample would be more proportional to the experimental error. Then the results would vary from zero to 100% flexibility with 100% flexibility corresponding to a Torsional Stiffness Ratio of one (1.0).

19. As I said before, at ERDL, the Gehman Tester is put inside the cold box for this test. This has required certain modifications in the apparatus. The dry-ice chamber, temperature controls and specimen rack were discarded. The specimens are stored in shelves in the rear of the box until tested. A bracket has been added to support the bottom specimen clamp. Pinch type paper clips have replaced the specimen clamps furnished with the instrument. This permits the operator to remove specimens one at a time from the storage shelf and place them in the clamps of the instrument, with tongs without bending or otherwise distorting the specimens. The specimen used is a T-50 specimen instead the specimen suggested by Dr. Gehman. We prefer this specimen because it eliminates a pronounced major axis in the cross section of the specimen and also the tab end eliminates errors due slight inequalities in the manner of clamping. The standard wise is of beryllium copper since the modulus of this wise is raised only 4% upon cooling from plus 73°F to minus 108°F. It should be noted at this time, that the standard wires furnished by the American Instrument Co. agreed with each other within 5%.

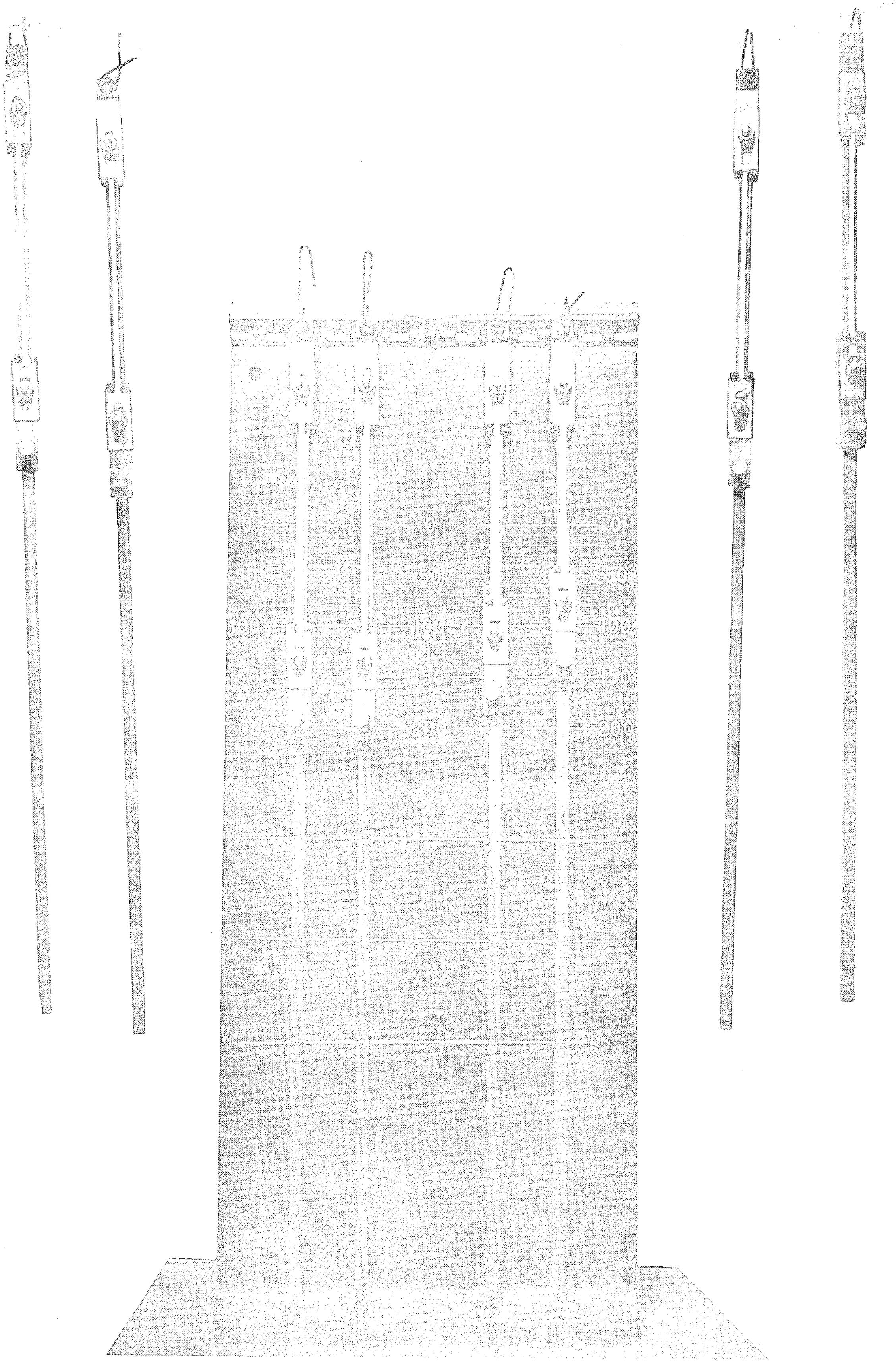
20. I stated above that the Torsional Stiffness Test is used for measuring increases in viscosity of rubbers. If the rubber is below its Second Order Transition Temperature it will give a very high Torsional Stiffness Ratio, well above 50 and therefore the accurate range of the tester. If the rubber has crystallized before testing, similar values will be obtained. Only rubbers which crystallize extremely readily well, however, give high values unless the rubber has been conditioned at the low temperature. Those that crystallize quite readily at the test temperature will show increased stiffness with increased time of conditioning.

21. The torsional stiffness test is required by the same specifications mentioned above. In these specifications we have required that the material have a Torsional Stiffness Ratio of not greater than 10.0 when tested at -65F after conditioning for 7 days at that temperature.

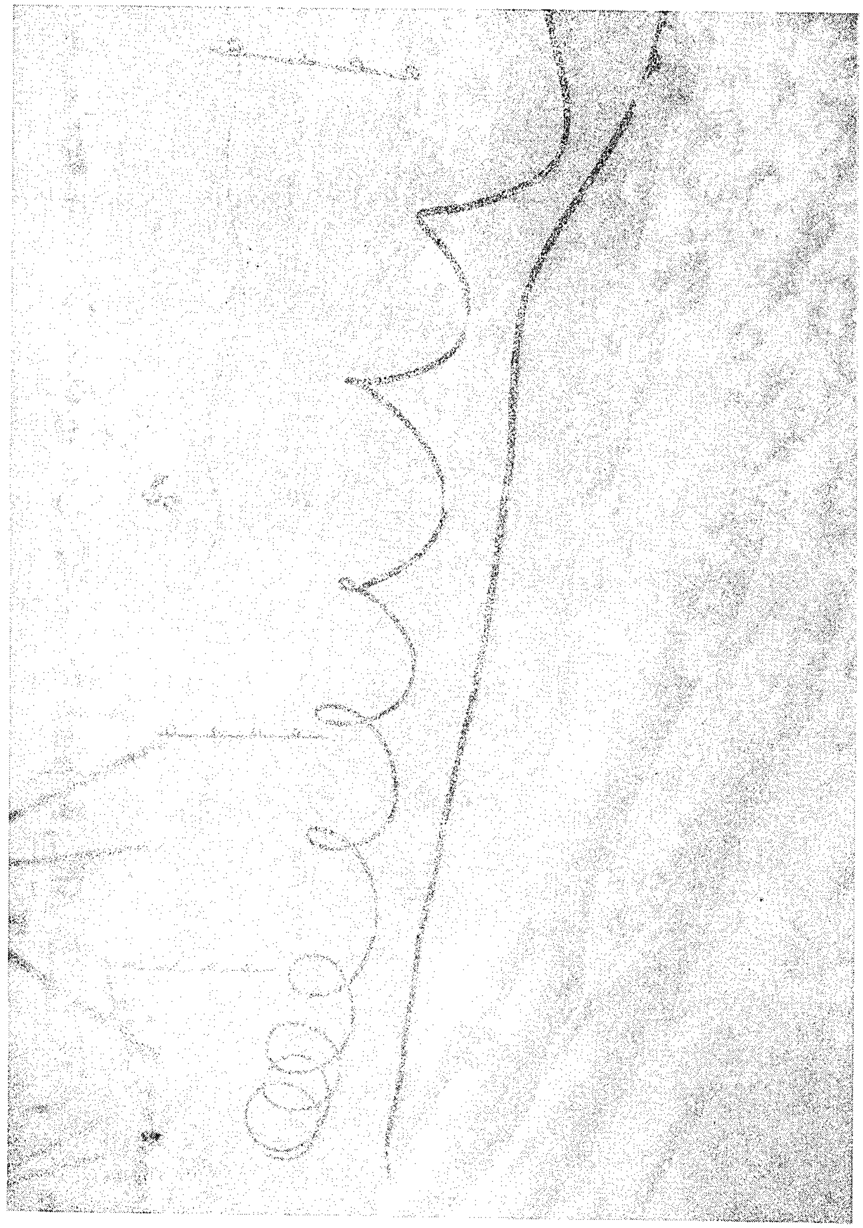
Slide 2

Slide 3

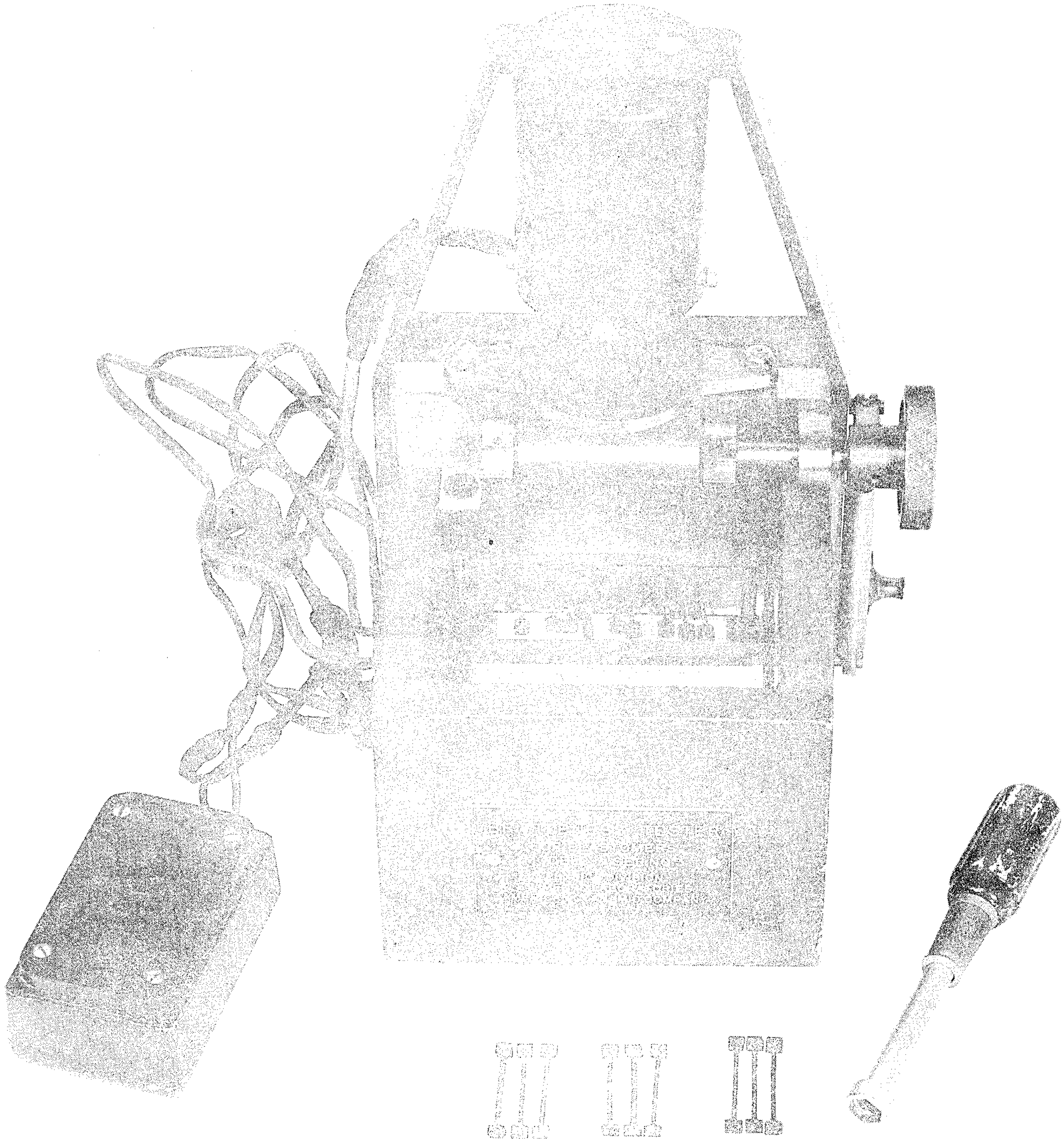
Slide 4



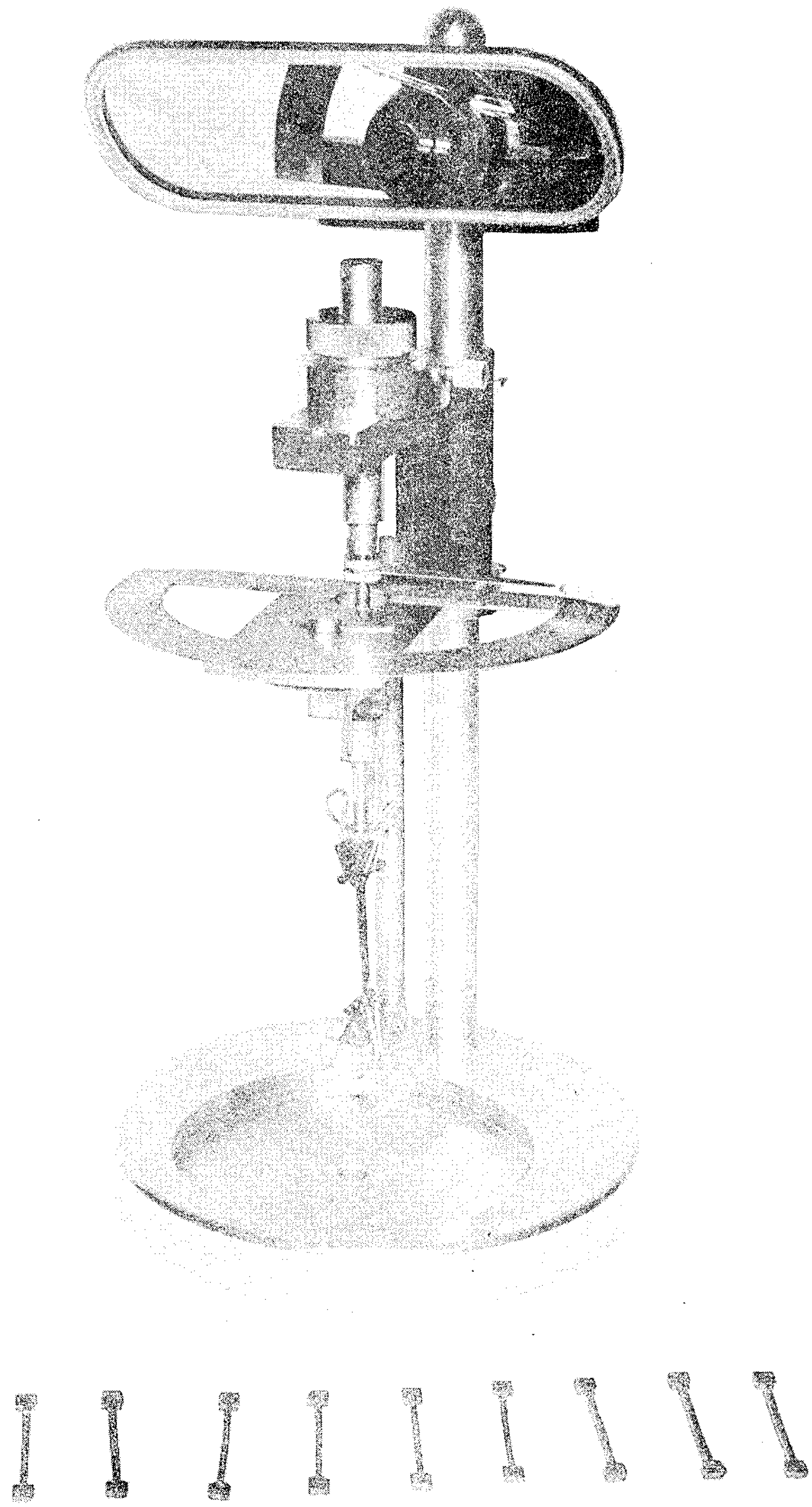
Slide 5



Slide 6



Slide 7



LOW TEMPERATURE TESTS USED BY THE

NAVY BUREAU OF AERONAUTICS

By

R. Harper

Naval Air Experimental Station

1. The Aeronautical Materials Laboratory has used, during the past few years, several of the newly developed low temperature test procedures including the Gehman, cold compression set and a modified low temperature retraction test. These tests will be discussed in detail later, but first the low temperature bending test known as the Thiokol Bent Loop Test used by the Bureau of Aeronautics in such specifications as MIL-R-6855 and MIL-R-5691 should be considered.

2. Although the Thiokol Bent Loop Test has some disadvantages, such as lack of control over the rate of deformation of specimens, it is difficult to visualize any other test to take its place in these specifications. Low temperature properties of fuel and oil resistant rubbers, when measured by the Gehman and cold compression set tests, are relatively poor. Fuel resistant rubbers conforming to Class I of MIL-R-6855 are compounded with Buna N polymers (Paracril 26 and Hycar OR25). Fuel soluble low temperature plasticizer is held to a minimum in order to meet the rigid volume change requirements. Class II material, for use with petroleum base lubricants requires general purpose neoprene polymers. Slide #1 lists Gehman and cold compression set data for typical Class I and II materials. Both stocks demonstrate excessive cold compression set even at -30°C. The Gehman values for the Class II neoprene material will

be misleading since first order transition effects are not produced in short term Gehman tests. With due respects to the valuable data provided by the Gehman and cold compression set procedures, both tests must be considered unsuitable for these materials. Their poor low temperature behavior is a function of the type of polymer used and restricted low temperature plasticizer content. There is little prospect of improvement in low temperature flexibility unless volume change properties are relaxed or a new polymer developed. With these materials, the bent loop test, even with its disadvantages, appears to be best suited. This test has been modified by the Aeronautical Materials Laboratory in an effort to eliminate some of the variables. The apparatus is basically similar to ASTM D736-46T. The moving jaw, however, is loaded with a dead weight of 50 pounds, and the load is released by pulling a key. No more than six specimens are tested simultaneously. Specimens are conditioned for five hours at -54°C. before flexing. Results to date have been reproducible using this modified procedure.

3. The Gehman Torsional Modulus, cold compression set and low temperature retraction tests have been used by the Aeronautical Materials Laboratory in compound development work. They have also been useful in explaining or predicting low temperature behavior of certain aircraft rubber parts. A modification of the low temperature retraction test has been written into a tentative specification for di-ester lubricant and low temperature resistant rubber material. The essential features of these three tests, as conducted by the Aeronautical Materials Laboratory are as follows:

GEHMAN TEST

The Gehman apparatus used by this laboratory is a modification of the equipment specified in ASTM D1053-49T. Slide #2 demonstrate this apparatus. Dry ice-acetone mixture is used as the coolant. The external cooling coil, A, is used to remove moisture from the incoming air and to provide some precooling. The cold dry air passes through a cooling coil contained in a dry-ice acetone bath, B. The cold air is piped to a 1/16" diameter orifice in the test chamber, D, where it is diffused through a thimble shaped fixture. Since the cooling air is under pressures up to 20 psi, additional cooling is produced by expansion of the cooling air through this orifice. Temperatures of minus 145°F, have been obtained with this equipment. Temperatures are controlled manually by varying the air flow. Slides #3 and #4 the test procedure for determining T_2, T_5, T_{10} and T_{100} values specified in ASTM D 1053-49T has been used for most of the work to date. Comparative tests on identical rubber stocks, conducted with the cooperation of Office of Rubber Reserve indicated good agreement between two laboratories using different types of equipment and between several operators. Some test work has been conducted also using the cold box procedure developed by the Material Laboratory, New York Naval Shipyard. The initial results indicated poor reports reproducibility but not enough tests have been conducted by this laboratory to determine its suitability. It is suspected that some of the difficulty may have been due to changes in the torsion wire on continued exposure to low temperature or accidental warming of the specimens while

installing them in the clamps. The Gehman test does not readily evaluate time effects associated with crystallization or plasticizer incompatibility. To do so would require excessively long storage periods at low temperatures. Gehman values alone are not indicative of low temperature performance. They should be supplemented with a crystallization accelerating test such as cold compression set or retraction tests.

COLD COMPRESSION SET

The Aeronautical Materials Laboratory uses the equipment shown in Slide #5 for this test. The general procedures described in MIL-R-900 are used. Percent deflection applied for various hardness grades is in conformance with ASTM D 395-49T for hot compression set. Tests have been conducted with this equipment at -30°C., -40°D., -54°C., and -65°C. after 22 and 94 hours conditioning. The accuracy and usefulness of this in predicting time effects are well established. It is probably one of the best tests developed to date for evaluating low temperature behavior of gasket or packing materials. It has a minor disadvantage in that specimens must be handled by forceps while being measured in the cold box. Occasional high results have been attributed to the operator accidentally touching the specimens with his gloves.

LOW TEMPERATURE RETRACTION

The Aeronautical Materials Laboratory uses a variation of the TR test developed by the Phillips Petroleum Company. Apparatus shown in Slide #6 is used. Four inch T50 specimens are elongated 50% and then exposed to low temperatures (usually -54°C.) for a period of 22 hours. At the end of this

time, the lower jaw is unlatched and the specimens allowed to retract. Percent retraction is observed on the scale 30 minutes after release. Most of the work to date has been conducted with nitrile rubbers containing high amounts of low temperature plasticizers. Slide #7 lists low temperature retraction, cold compression set and Gehman data for some of these materials. The retraction test correlates fairly well with the cold compression set 30 minute readings and Gehman values of most of these compounds. Not enough data has been accumulated with this test, however, to indicate its applicability to other polymers, particularly those that crystallize or show time effects. The test, although not as precise as cold compression set, has been reproducible thus far. It is probably the easiest and most simple low temperature test to perform and requires very inexpensive equipment that can be made in most laboratories. It provides an excellent indication of stiffening due to low temperatures. The test also accelerates effects due to crystallization, since the specimens are under tension. Data reported by Phillips Petroleum in the recent survey conducted by Office or Rubber Reserve indicated good correlation with cold compression set at -35°F.

4. It is believed that careful thought should be given to selection of the proper test method in evaluating a given material. Low temperature requirements should be kept within realistic limits. With present materials, good low temperature flexibility is obtained at the sacrifice of dimensional stability in fluids and general physical properties. Exaggerated low temperature flexibility require-

ments may result in generally inferior material. Wherever possible the low temperature test methods used should be consistent with service requirements for the end product.

5. Some thought should be given also, during this conference, to the question of low temperature conditioning. It has been customary to condition materials for a specified period of time at a given low temperature prior to testing. The Army, Air Force, and Navy Bureau of Aeronautics usually specify conditioning at -65°F. for periods of 5 to 168 hours depending upon the material being tested. It is generally recognized that time effects, influenced by first order transitions or plasticizer incompatibility, may take place at considerably higher temperatures. Future test procedures might be designed for conditioning at moderately low temperatures in the 0° to -40°F. range as well as at 65°F. and lower temperatures. Probably a great many of the so-called -65°F. flexible compounds are inferior to -40°F. stocks when stored for extended periods at temperatures in the -20°F. range. Certainly, temperatures in the 0°F. to -40° are frequently encountered in Arctic operations at ground level for prolonged periods. Shipboard requirements under the worst possible conditions also fall in this range. Future specification tests should be designed with these conditions in mind. Two or more low temperature conditioning periods may be required. An alternative would be to condition for several days at -20° or -40°F. and then decrease the temperature to -65°F. before conducting tests. Any discussion or information on this question would be welcomed.

TABLE 1 - LOW TEMPERATURE PROPERTIES - MIL-R-6855 MATERIALS

	Compound	Class I Grade 60 (Hycar OR25)	Class II Grade 60 (Neoprene W)
Gehman Torsional Modulus (Deg.C)	T_2	-11°	-32°
	T_5	-18°	-41°
	T_{10}	-20°	-43°
	T_{100}	-29°	-49°
Cold Compression Set after 70 Hours at -30°C (%)	Measured after 10 Seconds	97.4	97.9
	Measured after 30 minutes	89.1	97.9
Cold Compression Set after 70 Hours at -40°C. (%)	Measured after 10 Seconds	96.1	99.3
	Measured after 30 Minutes	96.1	98.7
Thiokol Bent Loop Test After 5 Hours at -54°C		Passes	Passes

TABLE 2 - LOW TEMPERATURE ROPERTIES - BUNA N COMPOUNDS

Compound No.		114	115	116	118	119	120	121	122	123	124
Low Temperature Retraction After 22 Hours at $-54^{o}C$(%)		45	20	25	33	3	5	30	5	28	5
Cold Compression Set After 22 Hours at $-54^{o}C$ (%)	Measured After 10 seconds	85.1	97.3	96.9	97.6	99.2	98.9	98.2	98.9	98.2	98.4
	Measured after 30 minutes	67.6	82.7	76.1	74.5	89.7	95.5	88.9	94.5	79.3	87.6
Gehman Torsional Modulus (Deg. C.)	T_{10}	51	44	47	48	38	41	45	41	45	41
	T_{100}	58	51	54	54	46	48	56	49	54	49

GENERAL DISCUSSION OF LOW-TEMPERATURE TEST WORK AT WRIGHT AIR DEVELOPMENT CENTER

By

Lt. Bernstein

(WADC)

At present, most of the low temperature tests which are employed by the Elastomer Plastics Branch of WADC are those which appear in Specifications. The most widely used are:

1. The ASTM Bent Loop Test, a general purpose test which appears in several specifications (such as, MIL-R-6855).

and 2. The ASTM Brittleness Test, another general purpose test (which appears in Inner Tube specifications).

Aside from their use in conjunction with specification testing, both of these tests are widely employed in our laboratories for evaluation of experimental compounds.

Some tests also used at Elastomers-Plastics Branch, which are more specialized, are:

1. Mandrel Tests for coated fabrics, in which the fabric is bent through 180° over a 1/8" mandrel after storage at -65°F. for a specified time.

2. The O-Ring jig test, which appears in several O-Ring Specifications (MIL-P-5516, MIL-P-5315A). Since this test is not widely known, perhaps a brief description would be wise. The o-ring to be tested is conditioned for 96 hours at -65°F. under no stress. During this storage period, it is suspended between two wedge-shaped supports mounted along the vertical diameter of the o-ring. At the end of the storage period, a weight (20#), which is attached to the lower wedge, is dropped. The resulting elongation of the o-ring

is measured after 30 seconds; the weight is then removed, and the recovery after 15 seconds is measured. This test probably doesn't tell much about the actual servicability of o-rings in installation, and better tests are needed.

3. The low temperature flexing tests which are used for hoses are more closely allied with service conditions. A typical example of this is a test included in MIL-H-6615. On the basis that the average man, in arctic conditions, can muster 60 pounds of pull, the specification states that a hose stored for 72 hours at -65:F. in a "U" shape shall require no more than a 60 pound force to bend it to a straight angle.

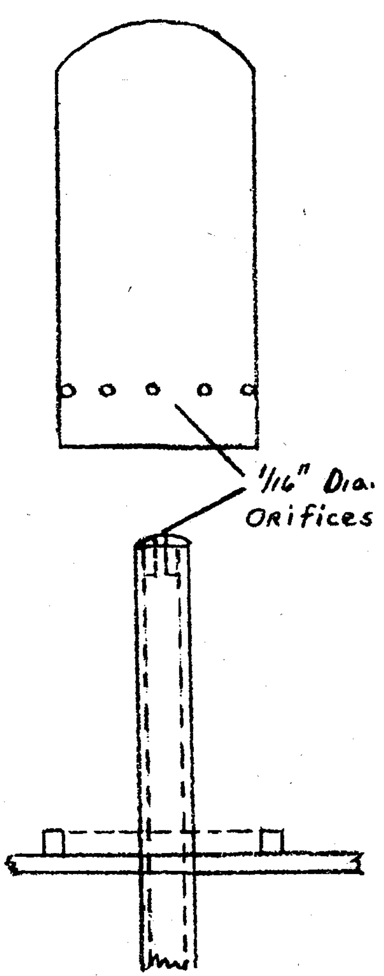
The tests which are also employed at Elastomer-Plastics Branch thought not as frequently, are:

1. Hardness - measurements of hardness of elastomers at low temperatures are used every once in a while for general evaluation of experimental compounds, and also for specification testing of silicone rubber under MIL-R-5847A. and 2. A test we call the "Finger Flex", which is a qualitative evaluation used in general lab work. It involves merely bending, with the fingers, a sample of the elastomer which has been stored at -65°F. The value of this test is questionable.

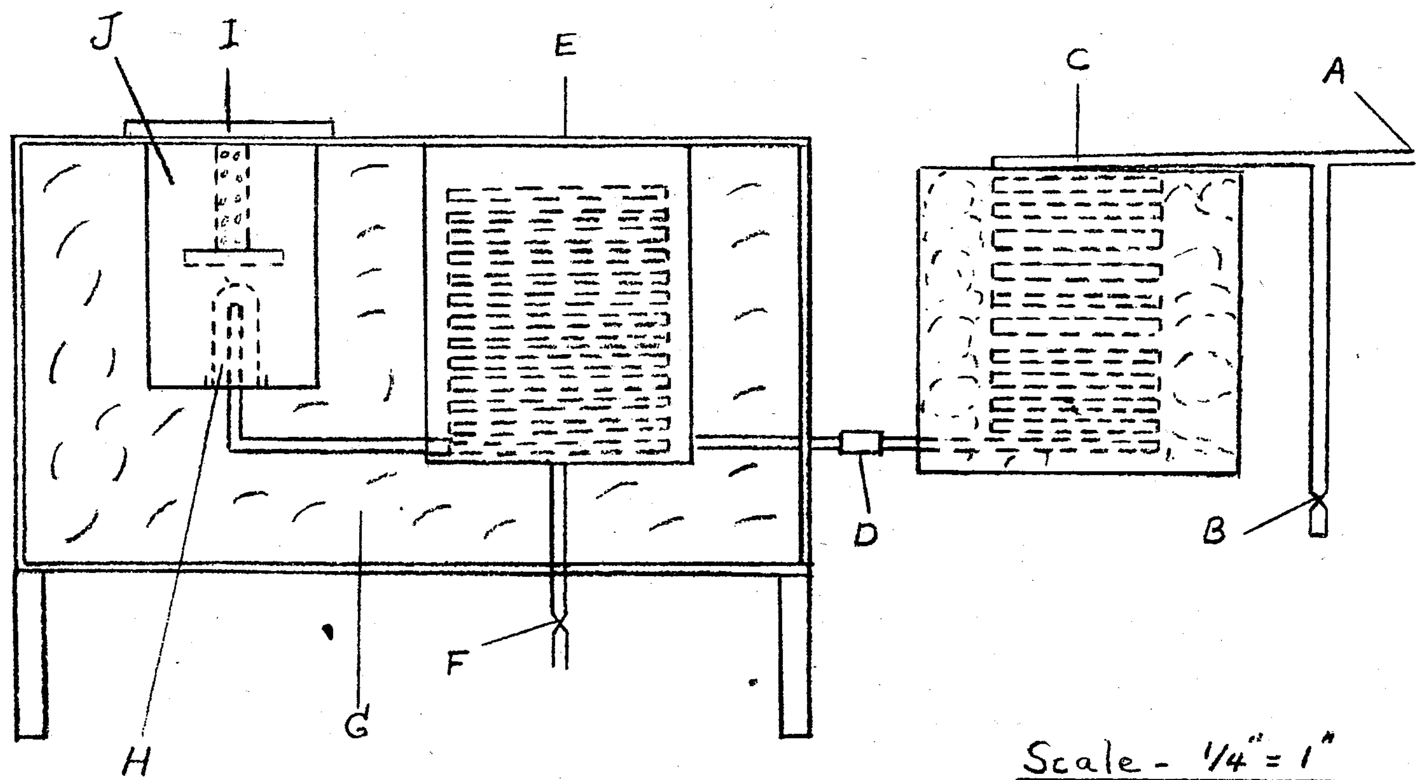
However, before describing our activities to rectify the deficiencies of our present low temperature testing, let me digress for a moment and describe two additional tests employed at WADC (not in the Elastomer Plastics Branch).

In o-ring testing, some of the other laboratories at WADC employs a specification test (MIL-P-5315A, and MIL-P-

Detail of H



(Drawn to scale)



Scale - 1/4" = 1"

A - Compressed air line

B - Adjustable hose clamp

C - Air-drying coil

D - Rubber tubing

E - Dry-ice-acetone bath

F - Drain

G - Fiberglas insulation

H - Jet orifice and diffuser

I - Specimen rack

J - Test chamber

Low Temperature
Cooling System
Adapted to Gehman
Apparatus 2/26/51

5516) which approximates service conditions. In this test, the o-ring is placed in a test jig where it acts as packing for fuel circulating at a minimum pressure of 15 psi. The entire system is placed in a cold chamber and the temperature is reduced from room temperature to -65°F. in increments of 10°. After each 10° drop, the stem of the jig, around which the o-ring is fitted, is rotated 24 times. To qualify, o-rings thus tested must show no sign of leakage.

The Tinius-Olsen Low Temperature Test apparatus has been used by Bendix in an Air Force contract. Apropo of this test, Pollack of Hardesty Chemical recently published details of an apparatus which employs the same basic principle as the Olsen tester. Pollack's apparatus measures the deflection (of a cantilever test sample) caused by a given bending moment. Similar to "bending beam" --correlation (w) Gehman type data when plotted on log-log scale, says Pollack.

WADC has, at present, initiated a low temperature test program in an effort to offset the deficiencies of the tests we now use. We have a Gehman low temperature test apparatus (the model sold by Aminco). Several difficulties were encountered in setting up this instrument for satisfacoty operation, and we have not yet gathered much data with it. A Gehman apparatus, designed after the modification used at the Government Laboratories at Akron, is under construction and should be completed within a month or so. We recently received a T-50 apparatus, which has been set up for T-R testing and much interesting data has been gathered.

* The Aircraft Lab, Hydraulics Lab, Equipment Lab.

In line with tests which are being readied for use by WADC is a problem which we would like to offer for discussion. Our High Polymers Unit has begun production of experimental new polymers, and we are faced with the problem of the testing of these experimental polymers. We need a good test for the evaluation of the crude materials - a test which will also be used by other labs on experimental polymers, to allow for comparison of data.

In deciding which tests to consider for overall-use in a long range low temperature testing program, WADC's thinking is probably not unlike that of other laboratories. It is the opinion, though of WADC, that if some concrete decision as to a standard series of tests are reached here, much needless duplication of effort can be avoided in the long run.

These standard tests should have the advantages of taking a short time to run, involve the use of a small sample, and be reproducible. Such a series of evaluations (chosen arbitrarily by WADC after weighing the literature) which may closely define the usefulness of an elastomer in service might include:

1. A Freezing Point Determination: This might be either a Gehman Torsional test, or a Temperature-Retraction test.

2. A Cold Compression Set test.

3. A Brittleness test; considering the two widely used Brittleness tests.

and 4. A Cold Hardness Test.

For any given elastomer, this entire series may not be neces-

sary to evaluate usefulness. However, in our estimation, they represent the optimum number of tests. And we feel that one test for each property <u>should</u> be chosen to produce a degree of uniformity of data. If, in time, new tests are developed which can produce a more valid correlation with service usage, they may be adopted. But at present, WADC feels that a definite series be adopted to allow for closer comparison of exchanged data.

We are not "stuck" with any one test, and will go along with the thinking of this group, providing conclusions are definite. We will undertake, for the group, any phase of testing considered necessary to provide a basis for standardization.

GENERAL REVIEW OF LOW TEMPERATURE TEST WORK OF ARMY ORDNANCE

By

R. F. Shaw

Rock Island Arsenal

The Ordnance Corps Rubber Laboratory at Rock Island Arsenal has been actively engaged for the past five years in the development of synthetic rubber compositions for Arctic service. The evaluation of these compositions by present military specification methods has been found to be of little value because of the inadequacy of the specifications in either stipulating the proper test method; in confirming the variables such as procedure, time, or temperature; or in prescribing a method which has little or no correlation with the service condition.

A literature survey was made during 1948 and a report entitled, "Test Methods for Elastomers at Extreme Low Temperatures" was written. This report classified all the basic types of low temperature measurements and gave illustrations and data pertaining to the important representative types such as brittleness, hardness, stiffness, and elastic recovery. This report was rewritten for publication and appeared in the INDIA RUBBER WORLD, July, 1950. Reprints of this report are distributed herewith.

In regard to the selection of test methods and the philosophy of their use, we should recognize three basic types:

1. Specification Methods

These may be representative of any of the basic

physical chara cteristics of rubber; should be completely standardized in every detail including apparatus, procedure, time, and temperature; and should preferably be of a go or no go type so that the results are not subject to misinterpretation. The use of a brittleness method which would indicate the absolute lower limit of usefulness would be representative of this type.

Example:

Brittleness -

Apparatus:	ASTM D746 (motor or solenoid)
Media:	Dry ice cooled air
Temperature:	Minus $65^{\circ}F$.
Conditioning:	One Hour
Report:	OK or failure (5 specimens)

2. Research Methods

These should be so designed as to allow control of variables such as applied stress, conditioning time, and temperature. This type allows one to explore the entire temperature range of interest, under varying conditions, and usually indicates an arbitrary point of limited usefulness such as increased stiffness.

Example:

TR (Temperature-Retraction) -

Apparatus:	U.S. Rubber or Phillips
Media:	Methanol-dry ice
Temperature:	Variable
Conditioning:	Variable
Stress:	Variable
Report:	Tabular data or curve

3. Actual or Simulated Service Tests

These are obviously dependent on the end item use. They may be actual tests in service mechanisms such as of brake cups or recoil packings, or they may be simulated such as mandrel bend tests for hose or wire and cable.

The test methods currently in use at Rock Island Arsenal for determining low temperature properties of elastomers are as follows:

Brittleness:

ASTM D736 - Bent loop - for existing specifications only

ASTM D746 - Motor Drive) for all revisions of specifications
- Solenoid)

Hardness:

Shore A Durometer - for existing specifications only

Shore D Durometer - for research methods

Admiralty Hardness Meter - for research methods

Elastic Recovery:

Compression Set - Proposed ASTM Specification

Temperature-Retraction - for research methods

Stiffness:

Torsion, Clash-Berg - ASTM D1043 for research methods

Service Testing:

Recoil mechanisms - Cycling tests at -65°F.

Equilibrators - Cycling tests at -65°F.

Brake mechanisms - Cycling tests at -65°F.

Boots, bellows, covers - Flexing at -65°F.

Gaskets - Sealing ability at -65°F.

It is considered that the basis of choice for a low temperature test method for rubber procurement specifications should be dependent on a apparatus simple in design and operation; should be reproducible between laboratories; should not be subject to misinterpretation but be of a go or no go type; and should be indicative of service conditions, i.e. one would not use a flexibility test to determine a compression gasket's efficiency.

Inasmuch as the Military Establishment is the largest user of Arctic rubber, it becomes essential that we present a definite, realistic approach to this problem by uniting our effort so as to present a solid front to such standardizing agencies as the Society of Automotive Engineers, the American Society for Testing Materials, and the entire rubber industry as well.

LOW TEMPERATURE TESTING OF RUBBER MATERIALS AT THE PHILADELPHIA QUARTERMASTER RESEARCH AND DEVELOPMENT LABORATORIES

By

C. B. Griffis

The most distinguishing property of rubber is its kinetic elasticity. Its ability for high extensibility and rapid recovery results from this property of kinetic elasticity. Many definitions of rubber are based upon this property. Schmidt and Marles in their book "Principals of High-Polymers, Theory and Practice", defined rubber as follows:

"To be considered a truly good "rubbery rubber", a material must meet the following criteria:

1. It must stretch readily and considerably under external tension.

2. It must possess a fairly high tensile strength and elastic modulus in the stretched state.

3. It must retract rapidly.

4. It must retract practically completely on release of tension.

The selection of a soft vulcanizate rubber material by the design engineer for any use in or on a piece of equipment is dependent upon these properties. Whether a golf ball or an automobile tire, the selection of rubber for the building material is dependent upon its easy extensibility and its rapid recovery. The very property of good wear in rubber tires is dependent upon this property. Where this property is not essential in building an item, rubber can be replaced by other materials. Thus we

have seen that many plastics are replacing rubber in such items as coated wire, coated fabrics, false teeth, and many molded toys.

The property of kinetic elasticity in rubber is highly dependent upon temperature, and particularly upon lowering the temperature. It is obvious that many soft vulcanizates that have high extensibility and rapid retraction at room temperature fail to exhibit these properties at -65°F or event at -40°F. How much any rubber stock loses these properties at low temperatures decides its use for the selected purposes at those low temperatures. Preliminary studies at the Phila. Quartermaster Research & Development Laboratories has shown a definite correlation between the loss in this property in rubber stocks with lowered temperature to increase in hardness, stiffness, and compression set, also changes in elongation, modulus, ultimate tensile strength, rebound, and abrasive resistance.

The desired properties of the rubber material for any particular use in an item can be established by certain requirements at room temperature. How much change from these requirements, resulting from temperature changes, can be tolerated and the item still have serviceable characteristics will have to be determined for each item. The quantative loss in the property of high extensibility and rapid retraction will determine the degree of change in the rubber material. There are several methods now being used by many laboratories for measuring quantatively the loss of this property. They include the U.S. Rubber Co. retraction test, the tension retraction test, and the compression set. Preliminary

studies have indicated that the U.S. Rubber Co. retraction test is the best test developed to date for measuring quantatively the change in the property of high extensibility and rapid retraction.

The Philadelphia Quartermaster Research and Development Laboratories has embarked on a program to study the possibility of using the U.S. Rubber Co. retraction test for performance requirements in Military and Federal specifications. Details of this test were published under the title "Retraction Test for Serviceability of Elastomers at Low Temperatures" by Smith, Hermonat, Haxo and Myer, Analytical Chemistry, 23, 322 (1951). The equipment used for this test is shown in Figures 1 and 2. The equipment and procedures used are essentially the same as those described in the above reference.

A series of rubber compounds were made using the following base polymers: Natural rubber, Neoprene FR, Hycar OR-15, Thiokol ST, Oil-extended polymers furnished by the General Tire and Rubber Co. and identified as General Tire Polymer "A" and Polymer "B", GRS-type rubbers representing charge ratios of Butadiene to Styrene of 90/10, 85/15 and 75/25 polymerized at 41°F, and GR-S-type rubbers representing charge ratios of Butadiene to Styrene of 95/5 and 85/15 polymerized at 122°F. The stocks were compounded to represent relatively the same state of cure from stock to stock.

Tests used for measuring loss in rubbery properties due to low temperatures for comparison with results obtained with the U.S. Rubber Co. retraction test are as follows:

1. Gehman Stiffness

The Gehman tests conducted are of two phases. The first phase is conducted with the equipment shown in Figure 3 and conducted according to the procedures described in ASTM designation (D 105349 T). The second phase is conducted with the equipment shown in Figure 4. The procedure used in this phase is as follows:

The torsional modulus of each sample is determined at room temperature ($73 \pm 3^{o}F$). The samples and the apparatus are then placed in a low temperature cabinet for conditioning at the selected test temperature. After the conditioning time and at the best temperature, the torsional modulus of each sample is determined. It is important to note that the sample is only twisted one time at the test temperature. The relative modulus at any temperature is the ratio of the Torsional modulus at $73 \pm 3^{o}F$ to the torsional modulus to that temperature.

2. Compression Set

The compression set tests are conducted with the equipment shown in figure 5. The tests are conducted according to the procedures described in Method B of ASTM Designation (D 395-49T) with the following exceptions:

Samples were deflected at $73 \pm 3^{o}F$ and then immediately stored at the storage temperature for the selected time interval. After the storage period and at the storage temperature, the compression set 10 seconds and 30 minutes after releasing the sample was determined.

3. Tension Recovery

The tension recovery test is conducted with the

equipment shown in Figure 6. Test procedures are as follows:

2 inch, T-50 samples are placed in the clamps and are then elongated 100 percent at $73 \pm 3^{\circ}$F. The clamps containing the elongated samples and the measuring board are then placed in a low temperature cabinet for a selected conditioning time and at a selected test temperature. At the end of the selected conditioning time and at the selected test temperature, the samples are released and the percentage of retraction after 10 seconds and 30 minutes is recorded. The percentage of recovery is reported and is calculated as follows:

$$\text{Percentage recovery} = \frac{E_1 - E_2}{E_1} \times 100$$

Where E_1 = Initial elongation of the sample

E_2 = The final elongation of the sample after releasing the sample in the clamp.

The cold temperature cabinet used is shown in Figure 7.

Gehman stiffness tests, compression set test, and tension recovery tests conducted at -65°F. for conditioning periods of 1 hour, 5 hours, 1 day, 7 days, and 28 days correlate very well with results obtained with the U.S. Rubber Co. retraction tests. The same conditioning periods will be repeated at -30°F and -50°F. Here discrepencies due to crystallization effects should be corrected.

Changes in hardness and brittleness due to temperature changes are being measured.

Procedures for measuring changes in elongation, modulus, tensile strength, rebound and abrasion due to temperature changes are being developed.

A method of determining the temperature below which the rubber stock no longer exhibits "rubbery rubber" properties has been developed using the U.S. Rubber Co. retraction test. This is called the 1 second TR-70 valve. It represents the temperature at which the rubber will retract 70 percent in one second when it is stretched 250 percent. Of the subject rubber stocks now under test, the TR-70 values and the 1 second TR-70 values are as follows:

	TR-70 minus °C	1-sec. TR-70 minus °C
Natural Rubber	9.7	9
Thiokol S^T	23.4	17
Hycar OR-15	4	2
Neoprene - FR	8.8	8
General Tire "A"	52.6	42
General Tire "B"	63.2	55
75/25 B/S @ 41°F	35	24
85/15 B/S @ 41°F	44.2	38
90/10 B/S @ 41°F	37.3	33
85/15 B/S @ 122°F	48	40
95/5 B/S @ 122°F	50	45

The General Tire Polymer "A" rubber stock is the only stock tested to date that has good "rubbery rubber" properties at -65°F. It is also interesting to note that this stock does not increase in stiffness after 28 days conditioning at -65°F. The compression set is only 30 percent and tension recovery is over 80 percent after the same conditioning.

From preliminary tests conducted, it appears that the U.S. Rubber Co. retraction test is an excellent method of

measuring kinetic elasticity properties of rubber materials; and that quantative measurement of kinetic elasticity properties is an excellent method for determining performance requirements for rubber materials subjected to low temperatures.

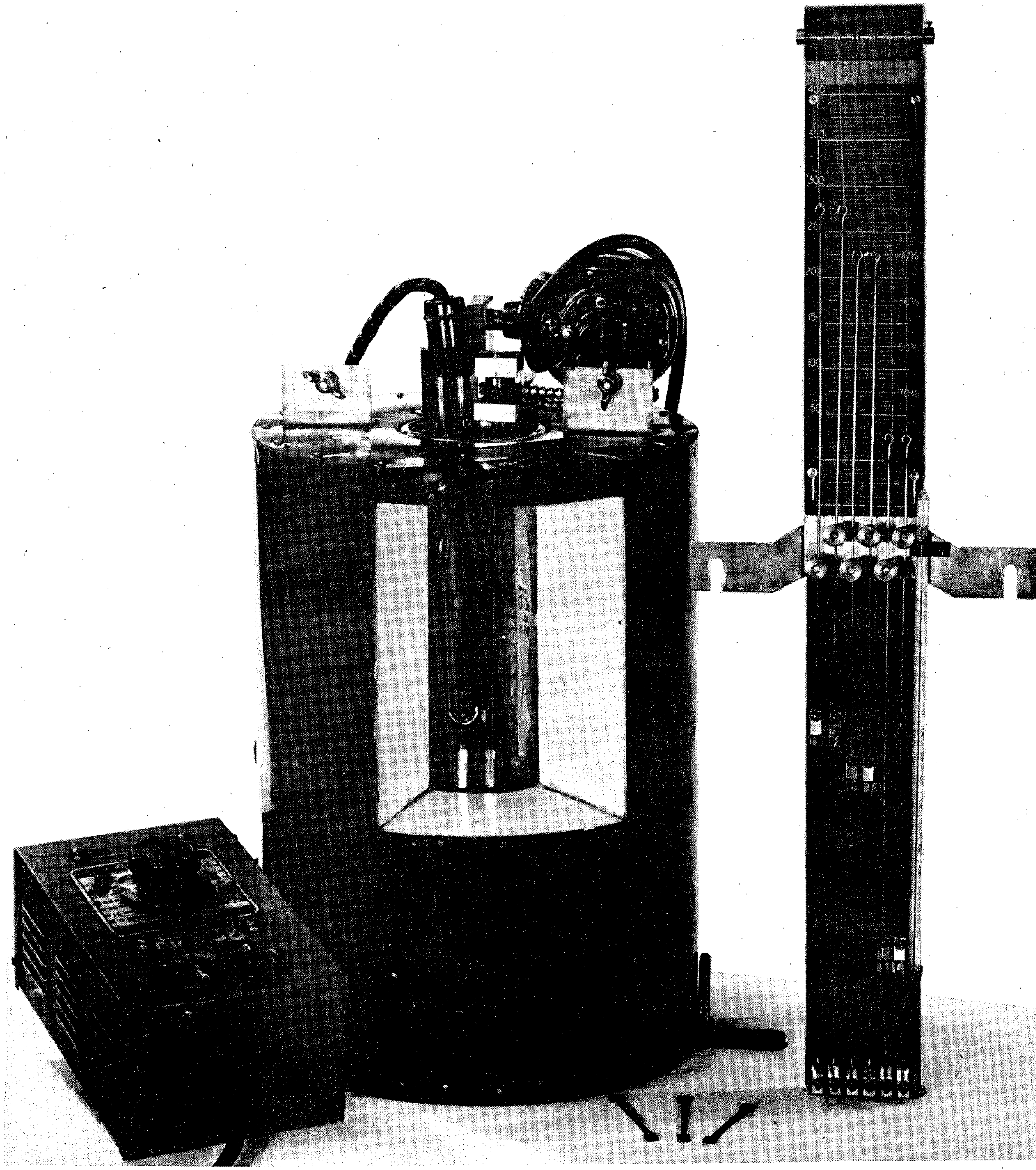


FIGURE I
U.S. RUBBER CO. RETRACTION TESTER

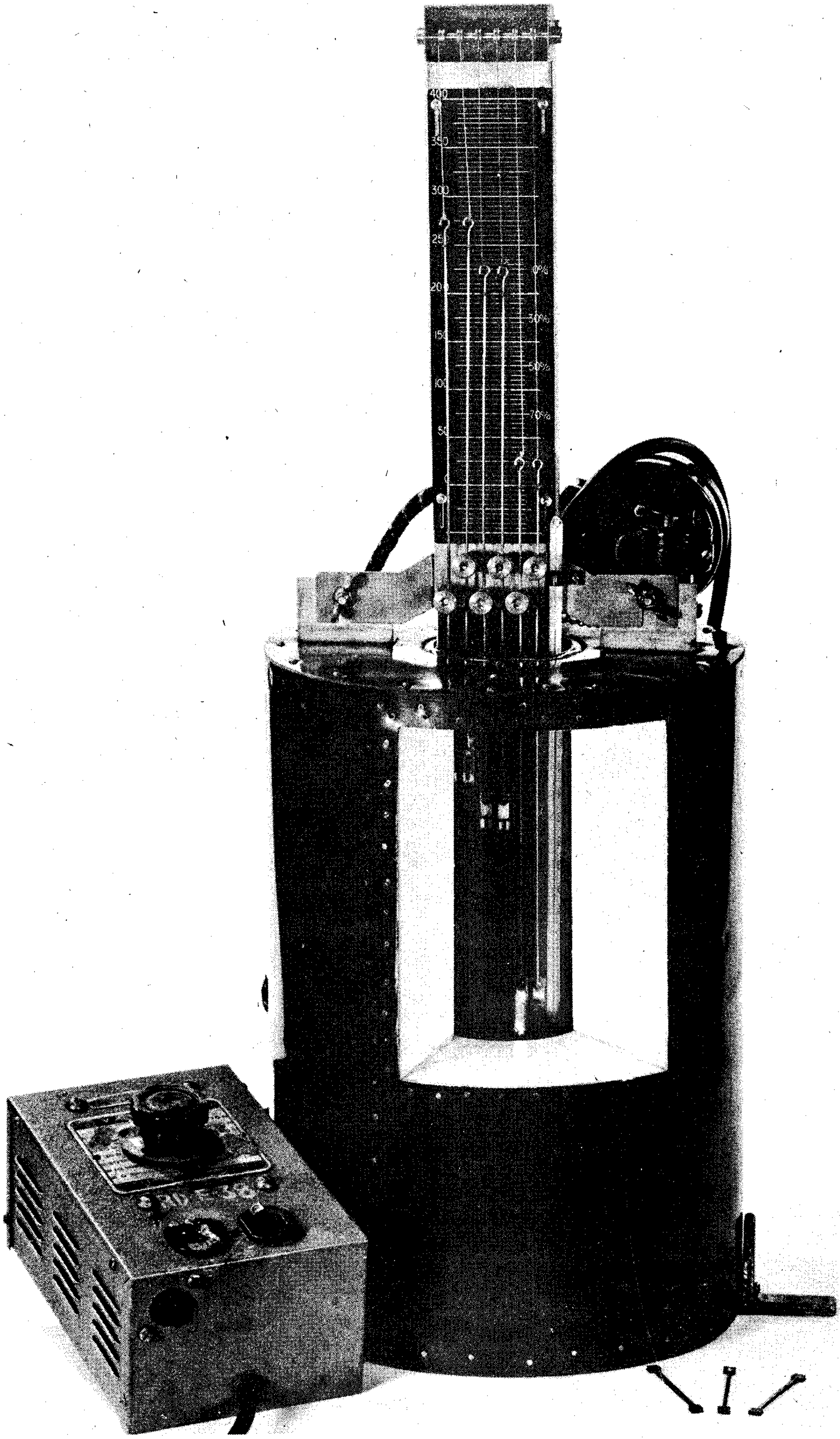


FIGURE 2
U.S. RUBBER CO. RETRACTION TESTER

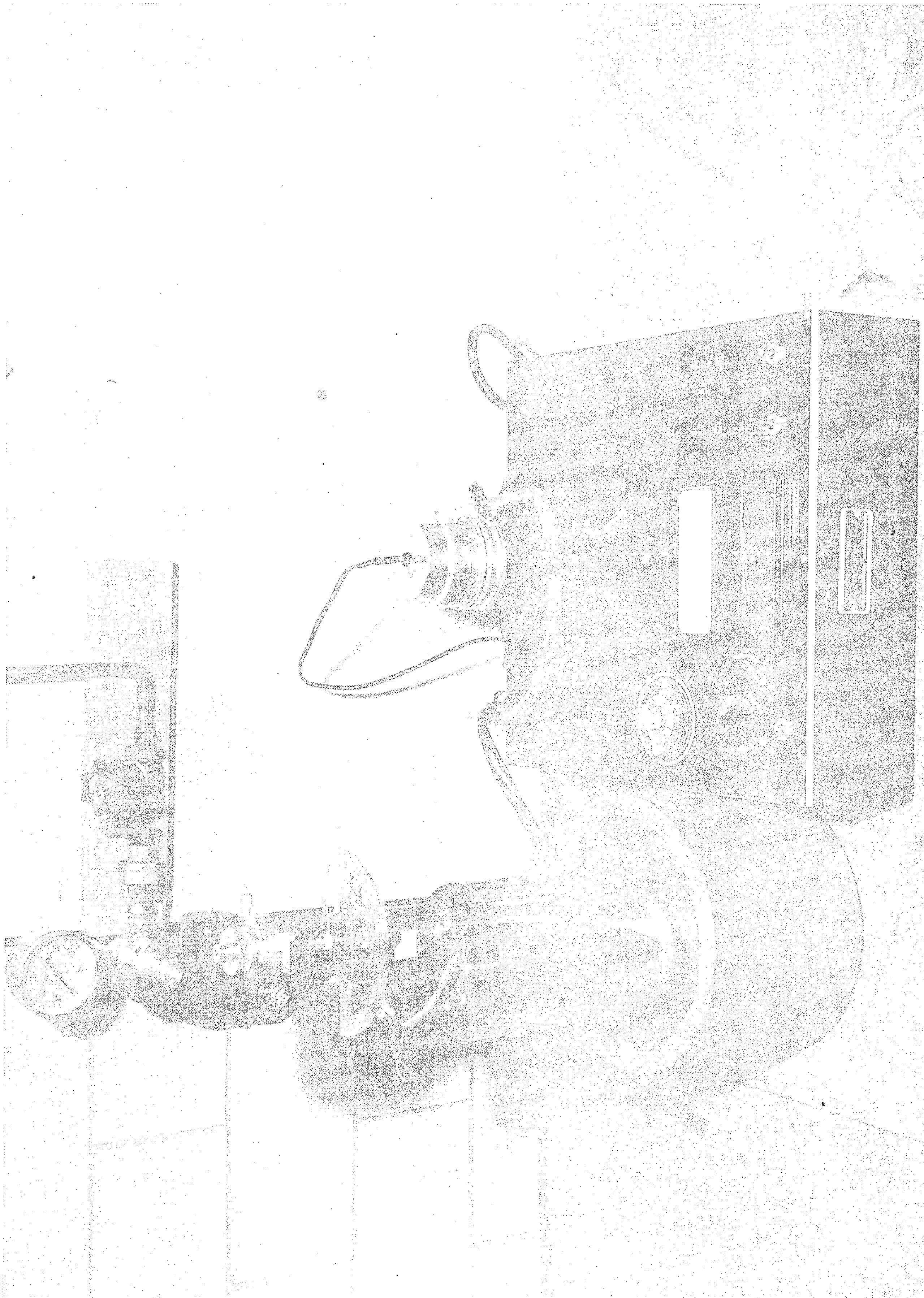


FIGURE 3
GEHMAN TESTER

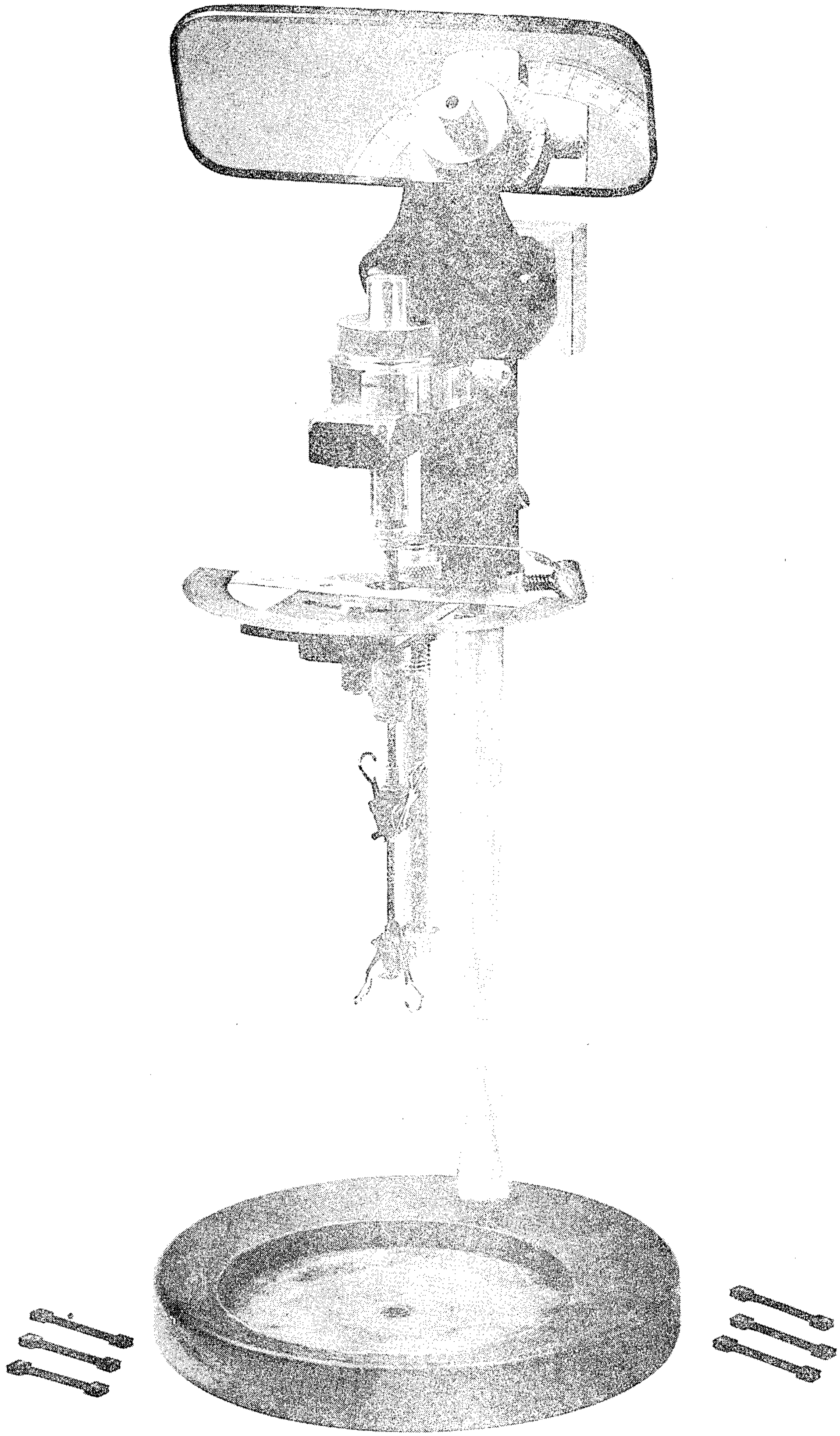


FIGURE 4
MODIFIED GEHMAN TESTER

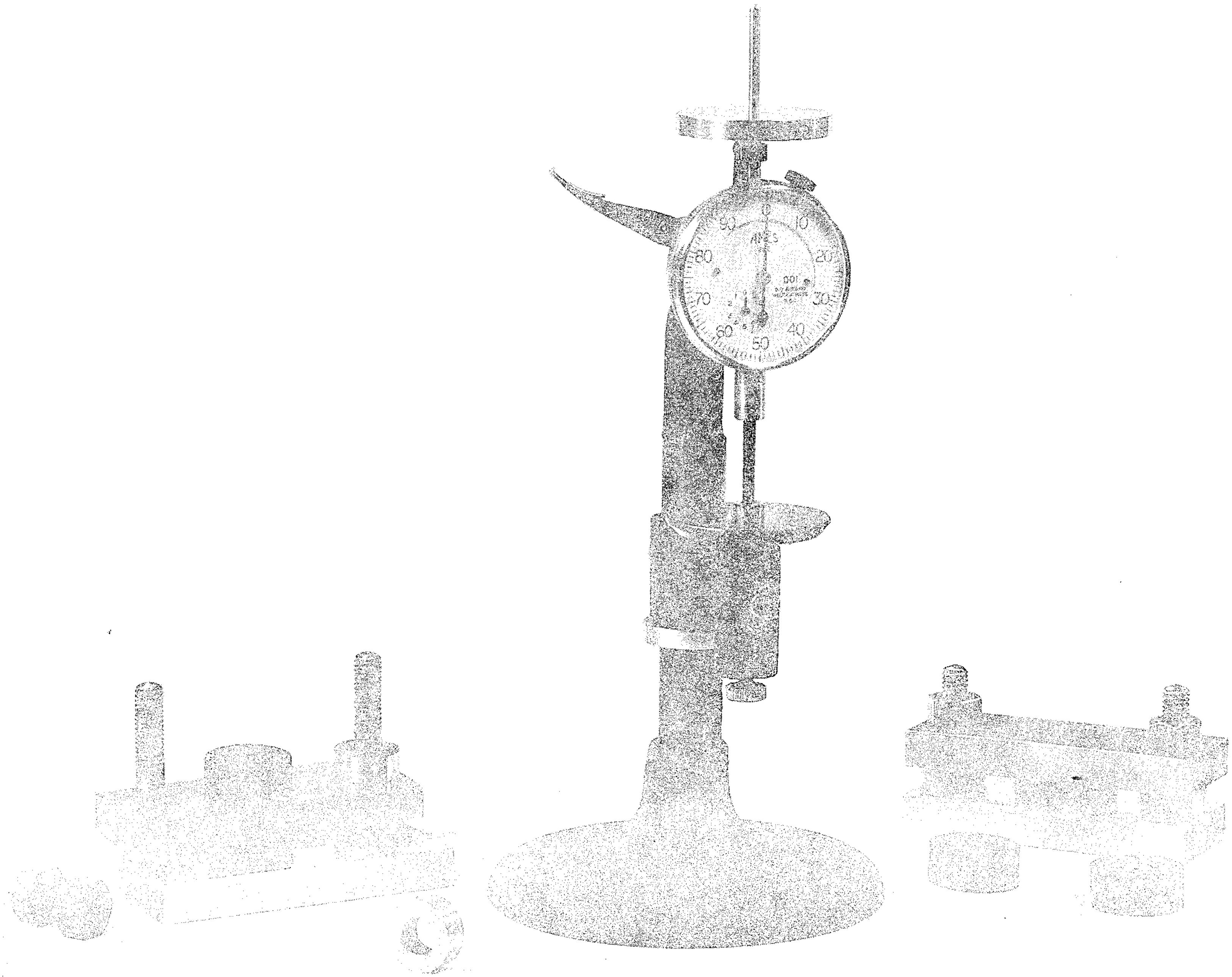


FIGURE 5
COLD COMPRESSION SET CLAMPS

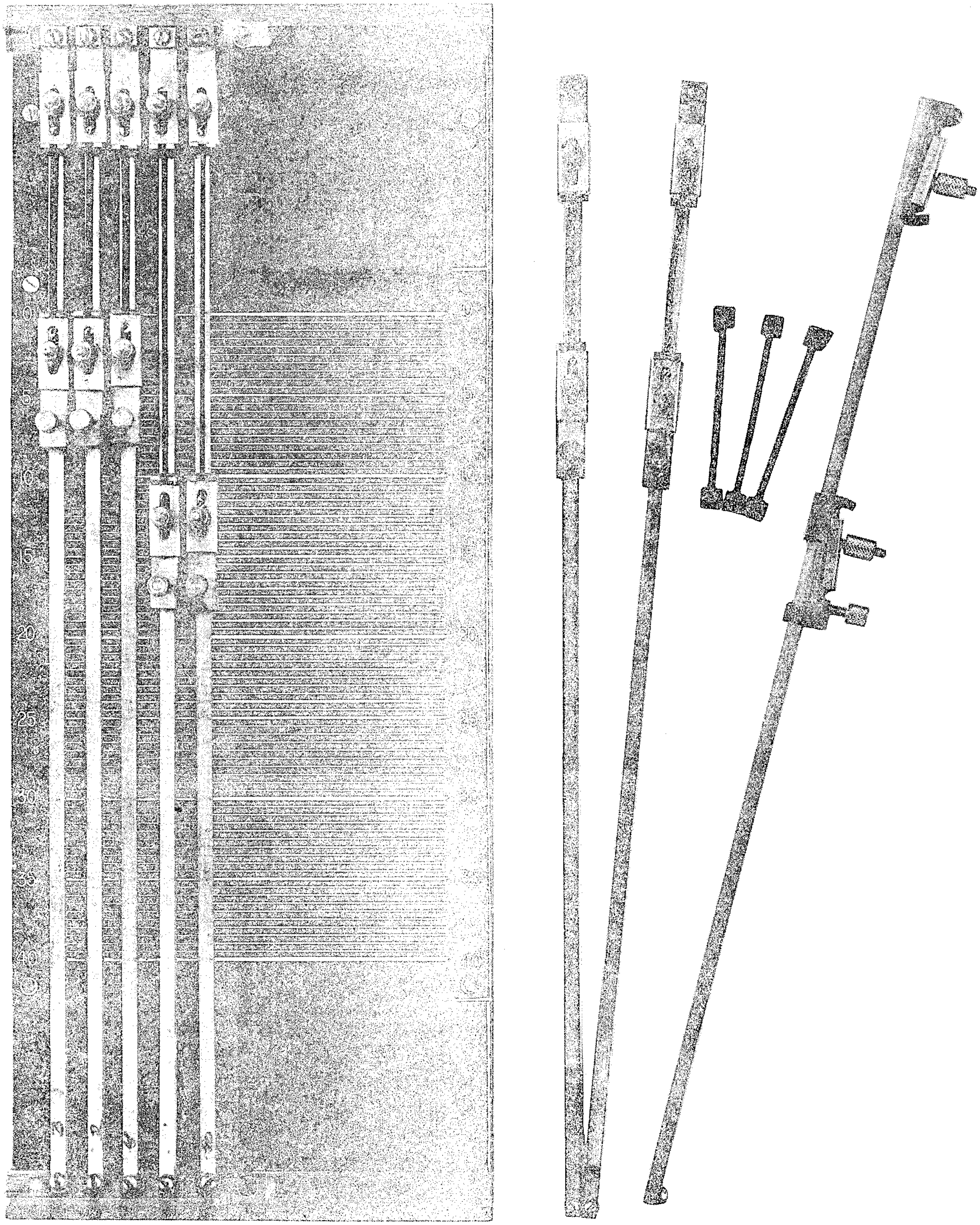


FIGURE 6

COLD TENSION RECOVERY TEST BOARD AND CLAMPS

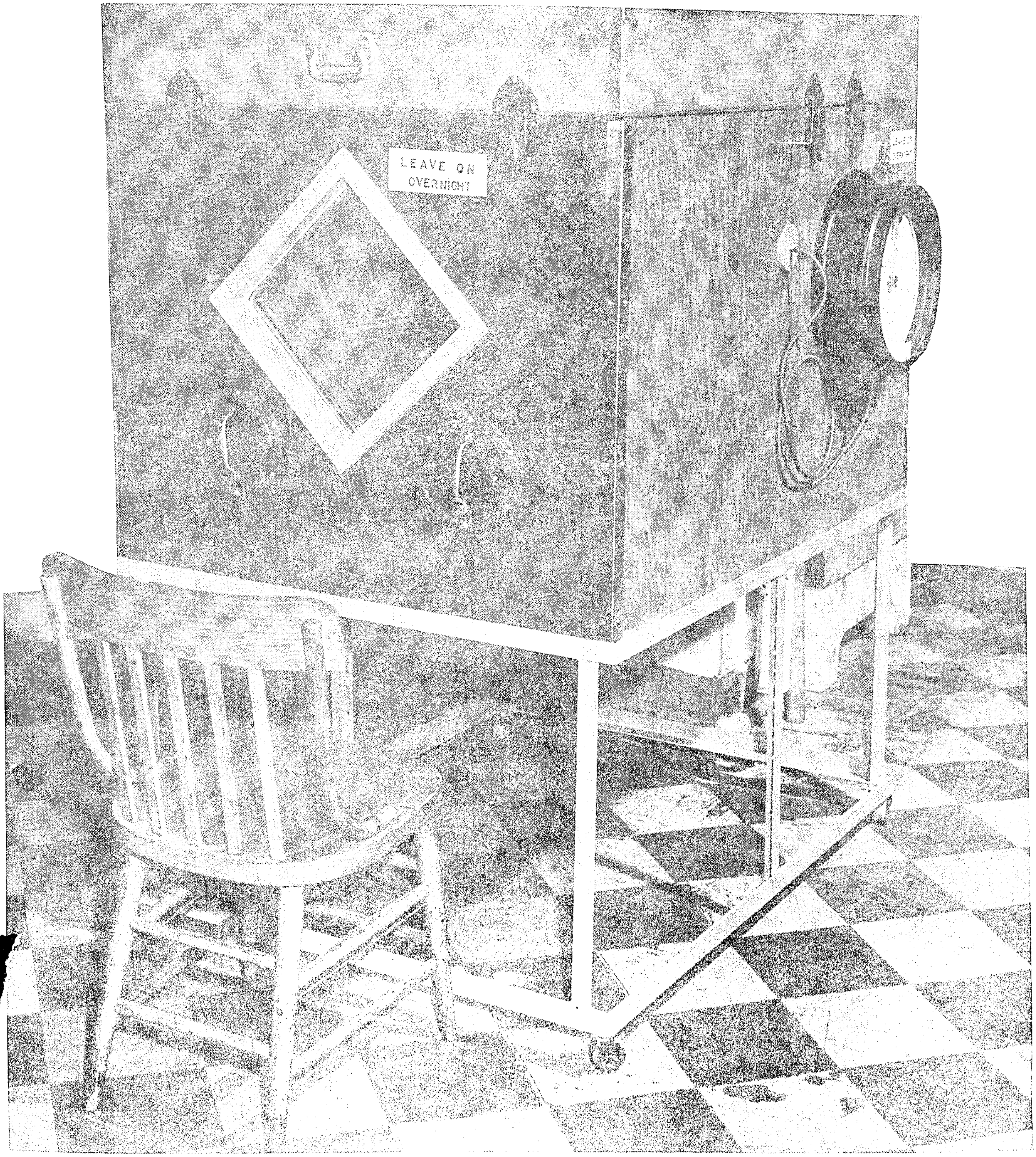


FIGURE 7
COLD TEST CHAMBER

DEPARTMENT OF THE ARMY
OFFICE OF THE CHIEF OF ORDNANCE
WASHINGTON 25, D.C.

ORDTB

3 April 1952

SUBJECT: Armed Forces Low Temperature Rubber Test Conference

TO: Engineer Research and Development Laboratory
Fort Belvoir, Virginia
ATTENTION: Mr. Philip Mitton
Materials Branch

Signal Corps Engineering Laboratories
Fort Monmouth, New Jersey
ATTENTION: Messrs. D. Lichtenstein, J. Lifland & W. J. Fontana
Squier Signal Laboratory

Detroit Arsenal
Center Line, Michigan
ATTENTION: Mr. J. E. Gaughan
ORDMX-ECR

Bureau of Aeronautics
Department of the Navy
ATTENTION: Mr. P. R. Stone
Airborne Equipment Section

Office of the Quartermaster General
ATTENTION: Dr. J. Montermoso
Chemicals and Plastic Branch
Research and Development Division

CO, Rock Island Arsenal
Rock Island, Illinois
ATTENTION: Mr. R. F. Shaw
Laboratory

CG, Wright Air Development Center
Dayton, Ohio
ATTENTION: Mr. E. Bartholomew & Lt. J. H. Bernstein
Materials Laboratory, Research Division, WORTE-3

Chief, Bureau of Ships
Department of the Navy
ATTENTION: Mr. T. A. Werkenthin
Code 344

CO, Naval Ordnance Laboratory
White Oaks, Maryland
ATTENTION: Dr. A. Lightbody

CO, Army Chemical Corps, Technical Command
Army Chemical Center, Maryland
ATTENTION: Captain M. Marks

Chief, Bureau of Yards and Docks
Department of the Navy
ATTENTION: Mr. T. C. Donnahue

Chief, Bureau of Supplies and Accounts
Department of the Navy, Arlington Annex
ATTENTION: Mr. T. J. Seery

1. On 4 and 5 March 1952, technical representatives of the various activities of the Armed Forces met at the Pentagon to discuss low temperature rubber tests. During the course of the meeting, it was agreed that it was desirable and feasible to limit procurement specification methods and apparatus to the following:

Brittleness:
Impact (motor or solenoid) ASTM D746 apparatus.

Hardness:
Indentor-dead load type (as in Federal Specification ZZ-R-601 which includes the Pusey & Jones and Admiralty testers).

Stiffness:
Gehman torsional Stiffness.

Elastic Recovery:
Compression set
Temperature-retraction
Tension-recovery.

2. Official confirmation of these agreements is requested to facilitate contemplated action of incorporating the above-mentioned methods and tests into rubber specifications as they are amended or revised by the cognizant agencies.

3. The minutes of the meeting on 4 and 5 March 1952, together with a proposed plan for future action, will be forwarded as soon as practicable.

BY COMMAND OF MAJOR GENERAL FORD:

R. W. White

R. W. WHITE
Assistant

R. I. A.
423/290
23 MAY 1952

ALL COMMUNICATIONS SHOULD BE ACCOMPANIED BY CARBON COPY AND ADDRESSED TO

TO INSURE PROMPT ATTENTION
IN REPLYING REFER TO:
O.O. NO. 423/298
ATTENTION OF
ORDTB-Materials

WAR DEPARTMENT
OFFICE OF THE CHIEF OF ORDNANCE
WASHINGTON, D. C.

20 May 1952

SUBJECT: Armed Forces Low Temperature Rubber Test Symposium, 4 and 5 March 1952

TO: Commanding Officer
Rock Island Arsenal
Rock Island, Illinois

1. Attached hereto are the official concurrences to agreements reached at subject symposium by the represented Armed Forces activities. It is requested that copies be included in the minutes of subject meeting.

2. Since Detroit Arsenal and your station have also expressed approval of the agreements reached, it is further requested that concurrence of the Ordnance Corps be indicated by including a copy of this letter as part of the minutes.

BY COMMAND OF MAJOR GENERAL FORD:

I. O. Drewry
I. O. DREWRY
Lt Col, Ord Corps
Assistant

Incl
Concurrences

4-3

DEPARTMENT OF THE ARMY
OFFICE OF THE CHIEF OF ORDNANCE
WASHINGTON 25, D. C.

1196

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R.I.A
423
229-Inc 1.
23 MAY 1952

Cmlre 337
00 337/1869

IN REPLY REFER TO:
ORDTB

5 May 1952

SUBJECT: Armed Forces Low Temperature Rubber Test Conference

TO: Commanding General
Research and Engineering Command
Army Chemical Center, Maryland

ATTENTION: Chief, Defense Materials Branch

Inclosed copy of correspondence, subject as above, is forwarded for concurrence as per telephone conversation of this date between personnel of your Command and Mr. I. Kahn of this office.

FOR THE CHIEF OF ORDNANCE:

R W White

R. W. WHITE
Assistant

1 Incl
Cy of letter dtd 3 Apr 52

CMLRE-D-3 337 1st Ind

Headquarters, Cml C Research & Engineering Command, Army Chemical Center, Maryland 14 May 1952

TO: Chief of Ordnance, Department of the Army, Washington 25, D. C.
ATTN: ORDTB

Concur.

FOR THE COMMANDING GENERAL:

Edgar C. ...

EMORY A. LEWIS
Colonel, Cml C
Deputy Commander

1 Incl
w/d

-wrc-5584

DISPOSITION FORM

SECURITY CLASSIFICATION (If any)

FILE NO.	SUBJECT		
QMGRJ 400.112	Armed Forces Low Temperature Rubber Test Conference.		
TO	**FROM**	**DATE**	**COMMENT NO. 1**
Chief of Ordnance Washington 25, D. C. ATTN: ORDTB - Materials	OQMG	14 April 1952	Montermoso 53013

400.112

5-893

1. Reference is being made to communication dated 3 April 1952 in connection with Low Temperature Rubber Test Conference held on 4, 5 March 1952.

2. This Office concurs with the methods of test and apparatus to be used which were agreed upon in this meeting.

FOR THE QUARTERMASTER GENERAL:

John R. Couture

JOHN R. COUTURE
Assistant Chief
Chemicals & Plastics Branch
Research & Development Division

NME FORM NO 96 1 OCT 48 Replaces DA AGO Form 897, 1 Oct 47, which may be used.

U. S. GOVERNMENT PRINTING OFFICE

4270

TECRD TSM
400.1 (8-93-01-001) (3 Apr 52) 1st Ind
SUBJECT: Armed Forces Low Temperature Rubber Test Conference

Engineer Research and Development Laboratories, The Engineer Center and Fort Belvoir, Fort Belvoir, Virginia 18 APR 1952

TO: Chief of Ordnance, Department of the Army, Washington 25, D.C.
ATTENTION: Assistant

These Laboratories concur with your records of the agreements reached at subject conference, as set forth in basic communication.

FOR THE COMMANDING OFFICER:

Don L. Burdette
DON L. BURDETTE
Executive Officer

1311

SIGEL-SMB-mf
Proj. 2005-M13-Rubber
(3 Apr 52)   1st Ind.   16 MAY 1952
SUBJECT: Armed Forces Low Temperature Rubber Test Conference
00. 337/1942
SCEL, Squier Signal Laboratory, Fort Monmouth, New Jersey

TO: Chief of Ordnance, Department of the Army, Washington 25, D. C.
ATTN: ORDTB

1. These Laboratories concur in the selection of apparatus for testing elastomeric materials at low temperatures, as listed in Paragraph 1 of basic communication, dated 3 April 1952. It is understood that these test methods are primarily for evaluating standard specimens, and do not preclude the use of other methods for determining the same properties in end-items or assemblies containing elastomeric materials.

2. Procurement specifications used by these Laboratories will be revised or amended as soon as the required test equipment is purchased and sufficient data are obtained to establish qualifying values under the test methods agreed upon.

FOR THE COMMANDING OFFICER:

for Herschel L. Stout
A. W. ROGERS, Chief
Components and Materials Branch
Squier Signal Laboratory

Asst. to Chief, C&M Branch, SSL, SCEL

HEADQUARTERS

Wright Air Development Center

WCRTE-3/JHB/jm

WRIGHT-PATTERSON AIR FORCE BASE

~~DAYTON~~ OHIO

IN REPLY ADDRESS BOTH COMMUNICATION AND ENVELOPE TO COMMANDING GENERAL WRIGHT AIR DEVELOPMENT CENTER ATTENTION FOLLOWING OFFICE SYMBOL

WCRTE-3

25 APR 1952

SUBJECT: Low Temperature Rubber Tests

TO: Office of the Chief of Ordnance
Attn: Mr. Irving Kahn
Department of the Army
Washington 25, D. C.

1. Reference is made to your letter dated 3 April 1952, concerning the decisions reached at the Pentagon meetings of 4 and 5 March on low temperature rubber tests.

2. This Headquarters confirms and approves of the selected low temperature tests. The value of this standardization is dependent upon how soon specific test procedures are defined.

3. With respect to standardized test procedures, this Headquarters has no comments on the Hardness, Stiffness, or Elastic Recovery tests, as long as definite procedures are promulgated as soon as possible. With respect to the Brittleness test, ASTM D746, it is suggested that minimum preconditioning time of 72 hours at test temperature be adopted. It might be well noted however, that this Headquarters is much in favor of the "Torsional Stiffness Ratio Test" (as used by the Engineering Research and Development Laboratories, Ft. Belvoir) being adopted as a specification test.

FOR THE COMMANDING GENERAL:

Richard R. Kennedy

M. E. SORTE
Lt. Colonel, USAF
Chief, Materials Laboratory
Research Division

402 112/6099

IN REPLY ADDRESS
COMMANDER
AND REFER TO NO.

WC:AL:fm

947

U. S. NAVAL ORDNANCE LABORATORY

WHITE OAK
SILVER SPRING 19, MARYLAND

8 MAY 1952

From: Commander, U. S. Naval Ordnance Laboratory
To: Chief, Office of Chief of Ordnance, ORDTB

Subj: Low Temperature Rubber Test Conference

Ref: (a) OCO ltr ORDTB to Various Addressees of 13 April 1952

1. Reference (a) reported that the conference on Low Temperature Rubber Testing held at the Pentagon on 4 and 5 March 1952, agreed that it was desirable and feasible to limit procurement specification methods and apparati for rubbers to four, one each for Brittleness, Hardness, Stiffness and Elastic recovery.

2. While the Naval Ordnance Laboratory has not used these recommended types of test equipment in their limited work on rubbers, it is planned to procure these and to use them in future elastomer work. We are quite in agreement with the proposed policy of standardization and, at the present state of knowledge, agree that the proposed test methods are probably most appropriate.

W. G. SCHINDLER

Albert Lightbody

Albert Lightbody
By direction

J2-6(344)
Ser 344-640

23 APR 1952

FIRST ENDORSEMENT on Ordnance ltr ORDTB of 3 April 1952

From: Chief, Bureau of Ships
To: Office of Chief of Ordnance
Department of the Army
Pentagon Building
Washington 25, D. C.

Subj: Armed Forces Low Temperature Rubber Test Conference

1. The Bureau concurs in the desirability of the recommendations contained in the basic letter. Revision of specifications on rubber products will be undertaken to reduce the test methods on brittleness, hardness, stiffness and elastic recovery to those selected at the conference of 4 and 5 March 1952.

973

DEPARTMENT OF THE NAVY
BUREAU OF SUPPLIES AND ACCOUNTS
WASHINGTON 25, D. C.

IN REPLY REFER TO
W21
A11/1
5 MAY 1952

A0 337/1896

From: Chief, Bureau of Supplies and Accounts
To: Chief of Ordnance
Department of the Army
Washington 25, D. C.

Subj: Armed Forces Low Temperature Rubber Test Conference

Ref: (a) OCO ltr ORDTB of 3 April 1952

1. The Bureau of Supplies and Accounts concurs in the agreement reached in the subject conference with respect to low temperature rubber tests as outlined in reference (a).

J H Taylor Jr.
J. H. TAYLOR, JR.
By direction

Copy to:
BuSandA (Code S4)
BuSandA (Code S33)
CSO, NSANY, Bklyn, N.Y.